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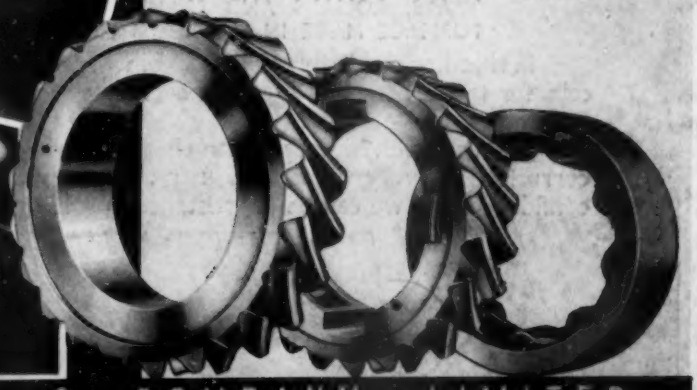

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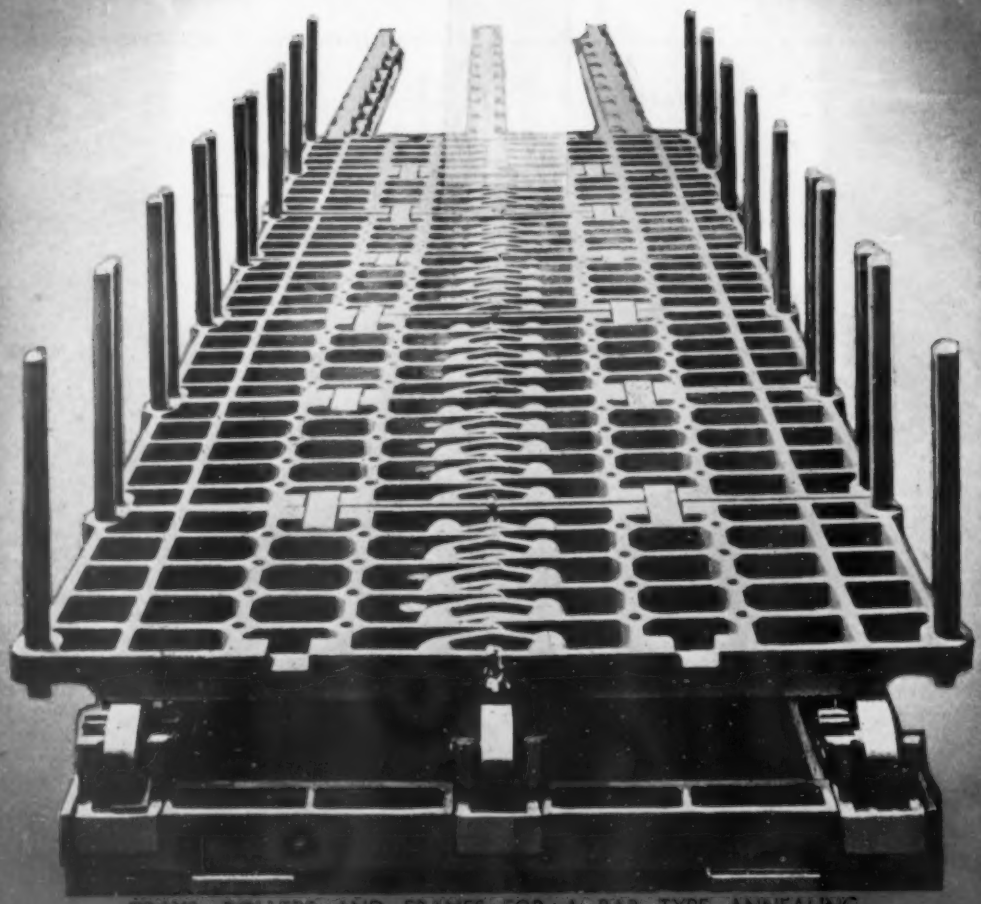

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## *The British Journal of Metals*

(INCORPORATING THE METALLURGICAL ENGINEER)


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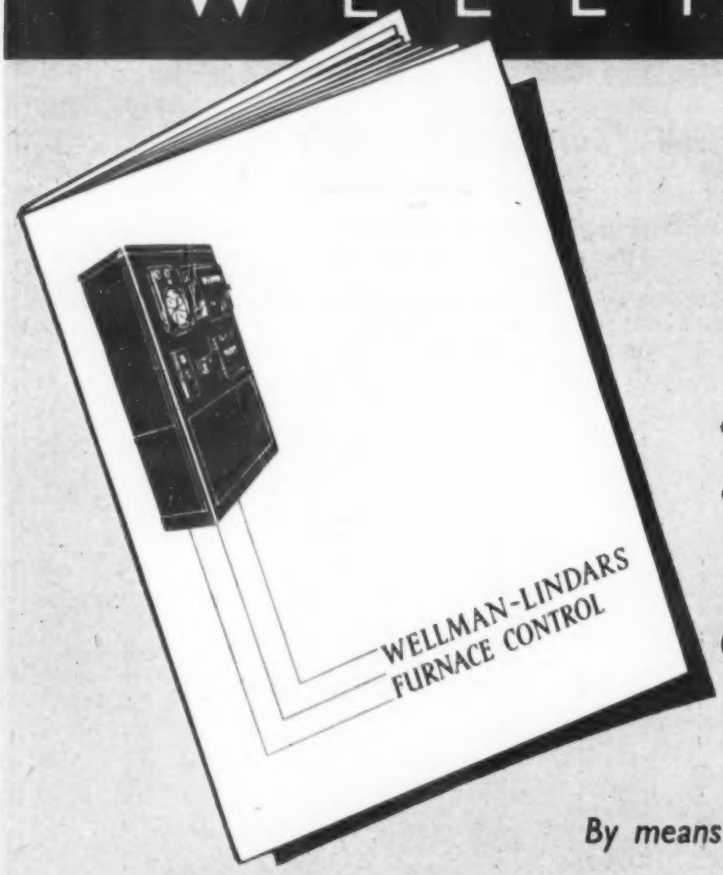


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METALLURGIA

THE BRITISH JOURNAL OF METALS.

INCORPORATING "THE METALLURGICAL ENGINEER."

NOVEMBER, 1946 VOL. XXXV No. 205

# High-Temperature Coking

UNTIL comparatively recently this country was very careless in its use of coal. It had abundant supplies, and the cost was so low that little thought was given to the efficient use of the coal. Excellent qualities were available for conversion into metallurgical coke in beehive coke ovens, on the Continent, however, good coking coals were scarce and much more attention was given to the efficient use of the grades available. While the beehive coking ovens were persisted with in this country, on the Continent, where these ovens were unsuitable, ovens of an entirely different design were ultimately developed which employed high temperatures for carbonising. Although beehive coking ovens continued to be employed in this country it was eventually realised that the best grades of coking coal are a wasting asset, and that the time would come when their use for purposes other than the manufacture of metallurgical coke would have to be restricted.

The change was effected gradually, and it was assisted by the increasing attention that was being given at that time to the preparation of materials constituting the blast-furnace burden. Screening out the fines and the reduction of lumps to a uniform maximum has proved of immense value, the extent of which has varied with the natural characteristics of the ores. Not less important, however, was the improvement in the manufacture of blast-furnace coke, resulting from a better knowledge of the types of coke of most economic value to the blast furnaces. Thus, concentration on the proper treatment of the ores coupled with the right kind of coke, of the right size and freed from fine dust and such particles of coke as are liable to create dust in the passage through the blast furnace, were contributing factors in developing high-temperature coking plants in this country.

The modern coke oven, employing high temperatures for carbonising coal has proved and is proving itself capable of producing a type of coke more suitable for the blast furnace than was possible by the older methods, and it is noteworthy that with high-temperature coking quite good coke is produced from coals formerly regarded as only of medium value. Indeed, the present technique of blending enables a fair proportion of coals to be used which were previously considered as unsuitable. Although the development of this method of carbonising coal has been concentrated in the last 40 years in this country, progress has been remarkably rapid, and the coal-processing operation has reached a high standard of efficiency. Much credit for this development is undoubtedly due to designers and operators of modern coke-oven plant, and Mr. G. A. Phillipson, in his presidential address at the recent meeting of the Coke Oven Manager's Association, was certainly justified in paying tribute to all those who had nursed the industry through its teething troubles, to reach the standard of efficiency not excelled in any other coal consuming operation.

While considerable progress has been achieved, Mr. Phillipson emphasised the fact that much remains to be done to further develop the potential wealth from coal through the carbonising process. An important problem which should continue to receive priority thought is the safeguarding of our coal resources, and the prevention of the waste of our most valuable coking qualities. It is true, that the industrial expansion of the country has been largely built upon coal, but it is doubtful whether it has been good for the future of the industry that this coal has been so cheap for such a rich quality. These two factors have enabled our industrial machine to compete freely in the worlds markets without resource to elaborate coal processing, but because of its cheapness its use has been abused, and too little attention has been given to its application. In consequence, research work in this country has not been maintained at standards comparable with those on the Continent.

To maintain our economic position and be in a position to continue to compete in the overseas markets, research organisations should be built up on the highest level. This fact is becoming the more apparent because of the increased price of coal and the shortage of supplies. The vigorous propaganda by the Ministry of Fuel and Power for the economic use of coal is an indication in this direction, and a nation-wide examination of coal-using methods is taking place, and already important economics have been established. This is of the utmost importance if we are to take advantage of our indigenous sources of power. It will also result in lowering manufacturing fuel costs by reason of reduced consumption. Whilst this policy is undoubtedly vital to the welfare of this country, it should be realised that it does not increase the value of coal as such. It is in complete processing of coal and the economic use of the resulting products that its value will be increased, and so enable the financial and economic structure of the mining industry to be maintained.

Apart from gas and coke credits, the modern coke ovens make most of the ordinary by-products by extracting and collecting the maximum of tar, sulphate of ammonia and crude benzole. The main problem is the production of precision quality metallurgical coke, and at the same time to control closely the quality and yield of these additional products. Mr. Phillipson, however, focussed attention on the subject of chemical utilisation and production of organic compounds from coke-oven gas. Of immediate importance to the welfare of the industry and of the country, he instanced water gas synthesis; ethylene recovery; thylox and similar systems of sulphur recovery; purification of, and recovery of phenols from, effluent liquor; and prevention of emission of sulphurous fumes from direct cooling

of gas. All these processes are largely adopted on the Continent and in other coal-processing countries. Mining as such is essentially a mechanical operation, whereas, the processing of coal is a chemical operation. Thus, the training for the two industries takes widely different channels, which cannot help but cultivate a differing mental approach to this immediate industrial question.

There is certainly a very pressing need for an adequate research organisation covering the processing of coal, and there should be complete separation from the mining industry. In this way encouragement would be given to an industry, which, under proper guidance could do much to obtain the maximum profit for this country's main raw material and result in higher standards of efficiency.

## Recruitment in the Ironfounding Industry

WHEN a full range of the products of the iron-foundry is considered it is somewhat surprising that there should be a difficulty in attracting potential moulders in sufficient numbers to keep the industry in a healthy condition. Thirty or forty years ago no such difficulty was experienced, although conditions in foundries then were far from being good. To-day iron-foundries are, on the whole, much improved from the point of view of the personnel, it is probable, however, that the difficulty arises from the fact that less craftsmanship is called for now than formerly. This does not mean that craftsmanship of a high order is not required but rather that it is in fewer hands. The trend towards quantity or mass production has brought its limitations in the application of skill by those who produce the castings. In the production of larger castings, however, which are more in the nature of one-off jobs, the need for individual skill remains at least as exacting as in former years.

Much attention has been given to means for stimulating recruitment for this industry by various organisations, all of which will welcome the announcement of the Government of a publicity drive. Certainly the Council of Ironfoundry Associations will welcome this announcement, because in recent months it has done much in the encouragement of young people to think about the possibilities of a foundry career. It has produced booklets, film strips, films, etc., and local organisations have arranged visits to foundries from schools, promotion of training schemes and various private incentives for recruitment purposes. The general question of increased amenities in making the industry attractive to labour has received further attention and regular progress has been made. Wages in the industry have been increased and in many sections are now at a very high point above the agreed level.

It is, however, not one or two, or all these factors combined which will result in attracting recruits for the ironfounding industry. It is the general propagation of the status of the industry which will achieve that purpose, and by raising it in the eyes of many at present prejudiced against it, will result in attracting recruits. One of the main reasons for the scarcity of recruits is the lack of appreciation which the industry has received as a craft in recent years. Whilst mechanisation has done away with a good deal of the craftsmanship required, it must be admitted that it can never be dispensed with entirely, having regard to the intricate castings required by industry, and, until some similar appreciation is given to the craft value of the industry at the present time, it can never attain the stature which will make it attractive to labour.

It is undoubtedly true that founding is not as attractive to boys as a number of other occupations in the engineering trades, and it is not easy to make them or their parents realise what interesting possibilities there are in the industry. Once the interest has been created in boys, however, it has been proved that, by the application of good apprenticeship methods, the interest can be maintained. But the greatest success has been, and, in view of the incidence of national service, will continue to be, with adult recruits entering ironfounding for the first time from the Forces or from other industries. These men conscientiously train under good industrial welfare conditions and become expressed advocates. When co-operation from the management and workers in individual foundries takes place, there is every hope for the future of ironfounding in the influx of such workers. To a great extent these are the people to whom the present publicity drive by the Government for ironfounding is directed.

We believe that much can be done to obtain suitable recruits by publicity methods and in overcoming the prejudice with which the industry is regarded by many. Unfortunately ironfounding is much underrated, and the status of the ironfounder suffers in consequence, but publicity is unlikely to effect a change in this direction, rather is it a duty for individual foundrymen and managements to promote that spirit which will gradually restore the prestige of the industry.

## Review of Swedish Technical Discoveries

REVIEWING the latest developments in physical science and engineering at the annual meeting of the Swedish Academy of Engineering Research, Professor Edy Velander, director of the Academy, referred to the Athoduc or "Flying Funnel," a propelling device for rockets, which would make it possible to send a projectile from, for instance, Europe to America in 30 minutes. An entirely new group of commodities of commercial possibilities was also mentioned—the radio-active isotopes. Especially interesting are the experiments to mark bacteria with the isotope C 14, a fantastic idea which opens up stimulating perspectives for the study of the spread of infectious diseases.

Many rare elements which previously have been regarded as scientific curiosities have in later years found practical use. Borax in small quantities in cast iron cylinder liners thus gives a fine graphitic structure. It is stated, Professor Velander said, that in pumps for certain chemicals castiron cylinder linings with 1% borax last nine times longer than ordinary linings.

Important improvements had also been made in the field of carbide metals. The further development in this field would perhaps make it possible for Sweden to use the rare earth metals found in the neighbourhood of Stockholm, which once gave renown to Swedish and Finnish chemists, when they succeeded in isolating them, for instance: yttrium, ytterbium, erbium and terbium, named after the localities where they were found.

# The Mechanical Properties, including Creep, of Aluminium Bronzes at Elevated Temperatures

By E. Voce,* Ph.D., M.Sc., F.I.M.

*(Communication from the British Non-Ferrous Metals Research Association)*

## Introduction

IT is a well-known fact that aluminium bronzes are pre-eminent among copper-base alloys in their resistance to oxidation and scaling at elevated temperatures. Data for the resistance to oxidation and scaling of aluminium bronzes in various atmospheres and in superheated steam have already been published[1] confirming the suitability of these alloys for use under normal conditions where the atmosphere or steam remains uncontaminated with such active impurities as sulphur dioxide, hydrochloric acid or chlorine.

On the other hand relatively little reliable information exists concerning the creep and other mechanical properties of aluminium bronzes at the elevated temperatures met with in steam and similar engineering practice, and the present work was undertaken in the Laboratories of The British Non-Ferrous Metals Research Association, with a view to filling this gap in knowledge. Tensile, notched-bar and creep properties were investigated.

As early as 1910 Rosenhain and Lantsbury[2] published the results of tensile tests on three aluminium bronzes at temperatures up to 550° C. The strengths which they gave for two 10% aluminium bronzes with and without 1% of manganese respectively were 22·7 and 24·0 tons/sq. in. at 400° C., figures which are appreciably higher than those found in the present work for similar materials at the same temperature. No subsequent publications on this subject, other than trade literature, have been traced.

Kanter[3] reported that die-cast aluminium bronze of 7·5% aluminium content gave a total creep of 1% in 10,000 hours at 290° C. under a stress of 4·5 tons/sq. in. and also at 450° C. under 1·3 tons/sq. in. Thum and Sigwart have communicated to the Deutsches Kupper-Institut the unpublished results of a few creep tests on three aluminium bronzes. The tests were only of 100 hours duration and stresses corresponding to total extensions of 0·5% or more at 300° and 350° C. were recorded. Since such large extensions in so short a time are of very little use in assessing creep characteristics for purposes of design, it seems pointless to include the results here. These are the only previous references to the creep of aluminium bronzes which have been found.

* Formerly of the British Non-Ferrous Metals Research Association. Now with the Copper Development Association.

1 A. F. C. Hallowes and E. Voce, METALLURGIA, **34**, pp. 95 and 119.

2 *Proc. Inst. Mech. Eng.*, 1910, p. 119.

3 "Symposium on Effect of Temperature on Metals," *Amer. Soc. Test. Mat.* and *Amer. Soc. Mec. Eng.*, 1931, p. 363.

## Metallographic Considerations

In order to appreciate the effect of temperature on the mechanical properties of aluminium bronzes, it is necessary to understand the changes of structure which occur when the alloys are heated and cooled.

Part of the copper-aluminium equilibrium diagram, as edited by Raynor for the Institute of Metals, is reproduced in Fig. 1. From this it will be seen that immediately below the solidus a 10% aluminium bronze consists wholly of the $\beta$ phase. On cooling to temperatures below 840° C. however, there is a separation of $\alpha$, which increases as the temperature is reduced to the eutectoid horizontal at 565° C. This $\alpha + \beta$ type of structure, being strong and tough is that usually preferred for engineering purposes, and is designated the "normal" or "normalised" material in this paper. It can be retained by quenching or moderately rapid cooling from temperatures between 565° and 840° C. Air cooling from temperatures above 840° C. also gives the $\alpha + \beta$ structure, because the separation of $\alpha$ from $\beta$ takes place quickly; the unchanged $\beta$ phase can only be retained by rapid quenching from temperatures near the solidus. It is evident, therefore, that the desirable "normal" structure can easily be

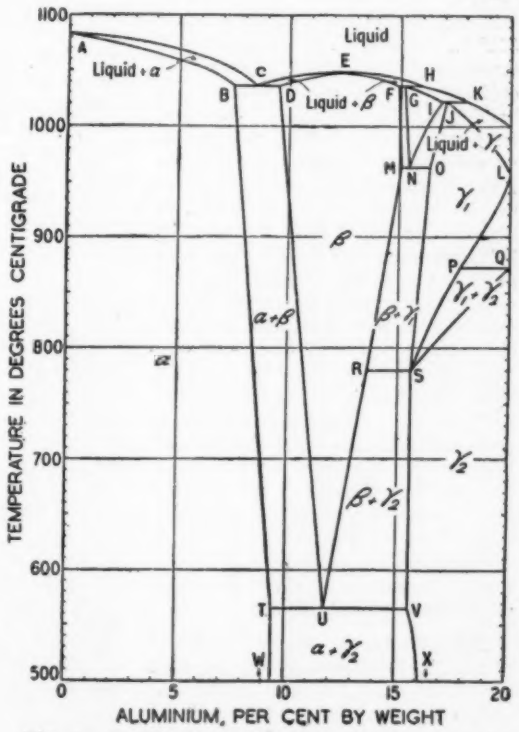


**Fig. 1.—Equilibrium diagram of the system aluminium-copper.**

**Fig. 2.—Normalised 10% aluminium bronze. × 200.**

**Fig. 3.—Stabilised 10% aluminium bronze. × 200.**

obtained in practice in several different ways, and it is in this condition that the bulk of 10% aluminium bronze is put into service. Fig. 2 shows the structure of a straight 10% aluminium bronze in the "normalised" condition. The $\alpha$ is light in colour while the $\beta$ shows the acicular markings typical of this constituent.

Though theoretically unstable below 565° C., the $\alpha + \beta$, or "normalised" structure, is retained indefinitely at ordinary temperatures. On the other hand, at the moderately elevated temperatures with which this paper is concerned the $\beta$ breaks up into an intimate mixture of $\alpha$ and $\gamma_2$ at a rate which increases as the eutectoid horizontal at 565° C. is approached, being rapid at 500° C. but quite slow at 250° C. Thus, whether attained quickly or slowly, the final stable structure at such temperatures consists of $\alpha + \beta$, but of $\alpha + (\alpha + \gamma_2)$. For present purposes "stabilised" material is that in which the reaction $\beta \rightarrow (\alpha + \gamma_2)$ has proceeded to completion, either through a preliminary heat treatment at, say, 500° C., or as a result of exposure to elevated temperatures in service. The stabilised type of structure is shown in Fig. 3, which should be compared with Fig. 2, noting the change in the areas which originally consisted of $\beta$.

The presence of the $\gamma_2$ phase in aluminium bronzes is usually regarded as detrimental to the mechanical properties; it has a tendency to cause brittleness and to diminish ductility, especially if the general texture of the structure is coarse through overheating. For engineering purposes at ordinary temperatures care is generally taken to avoid formation of $\gamma_2$, but it is clear from what has been said that any such precaution is foredoomed to failure where service at elevated temperatures is involved, for the $\beta$ will inevitably break up into $(\alpha + \gamma_2)$ at a rate depending upon the temperature concerned. Thus, at the outset of the present work a major problem was to ascertain to what extent, if at all, the unavoidable formation of $\gamma_2$ was, in fact, detrimental from the point of view of service at elevated temperatures. The experimental work about to be described, demonstrated that a "stabilising" heat-treatment, leading to the formation of $(\alpha + \gamma_2)$, actually improved the resistance to creep.

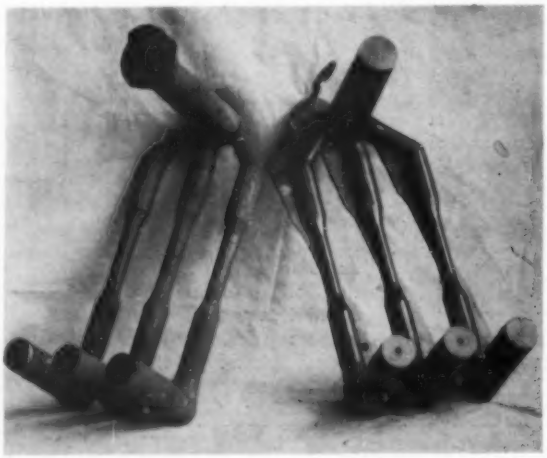

**Fig. 4.—A casting and the pattern.**

## Testing Procedure

Ordinary tensile tests were carried out at room temperature, at 250°C. and at 400° C., creep tests at 250° C. and at 400° C., and notched-bar impact tests at room temperature, 200° C. and thence at intervals of 50° C. up to 600° C. The maximum of 400° C. was adopted for the tensile and creep tests because this is approximately the highest temperature likely to be encountered in steam engineering and similar practice, for which aluminium bronzes would appear to be useful.

Ten aluminium bronzes of different composition were investigated under various conditions of heat-treatment, but by no means all the tests enumerated above were applied in every case. In particular, it was considered preferable thoroughly to explore, under different conditions, the creep properties of three selected alloys than to carry out fewer tests on a wider variety of materials.

The testing methods are briefly described below.

*Tensile Tests.* The tensile tests were performed upon a 20-ton Amsler machine (5 or 10 ton range) operating at a strain rate kept approximately constant at about 0·025 per minute. The parallel portion of each specimen was 2 in. long with a sectional area of 0·1 sq. in. (0·357 in. diameter), and two overlapping gauge lengths of 1·25 in. ($= 4\sqrt{\text{area}}$) were marked upon it. For the tests at elevated temperatures a resistance furnace was used, so wound that the temperature gradient along the length of the specimen could be controlled and kept to a minimum. Thermocouples at the top and bottom of the gauge length did not differ by more than 2° or 3° C°. About 30 minutes were needed to adjust the temperature, which was then maintained constant at the required value for a further 30 minutes before beginning to apply the load. Depending on the

ductility of the specimen, pulling occupied from 5 to 25 minutes, during which time the temperature was kept as constant as possible, though the mechanical work being performed upon the specimen often caused a slight rise which was difficult to check.

*Notched-Bar Impact Tests.* For these experiments a standard 120 ft. lb. Izod machine was fitted with a striker to deliver the blow in the Charpy manner to a specimen resting as a beam on a specially designed anvil. The specimen measured 1 cm. × 1 cm. × 6 cm., and carried a standard Izod notch at the centre of its length on the face opposite to that struck by the hammer. A batch of specimens was brought to the required temperature in a resistance furnace in close proximity to the anvil and held thereat for 30 minutes before testing commenced. One operator transferred a specimen from the furnace to the anvil, whereupon another released the hammer. The specimen was broken in an average of 1·8 seconds from the instant of leaving the furnace and experiment showed that the effect on the notched-bar value of the temperature drop in this short interval could be neglected. After each specimen had been withdrawn, the temperature of the furnace was re-adjusted before another was taken.

Though the results should not be compared directly with orthodox Izod or Charpy values, they are comparative among themselves and duplication was normally to within ± 2 ft. lbs., greater scatter than this indicating instability of structure under the conditions of test.

*Creep Tests.* The creep tests were conducted on machines of the orthodox type, in which the load was applied to the specimen by means of weights acting through a lever. The specimens had a parallel length of 5 in. with a sectional area of 0·1 sq. in. Extensometer readings could be reproduced to within ±0·0001 in., so that the strain on the specimen could be measured to $\pm 2 \times 10^{-5}$. Thus, when the creep rate remained constant over a period of ten days or more, as was usually the case, it was quite possible to determine this rate with reasonable certainty to a lowest value of $2 \times 10^{-6}$ per day, and the apparatus therefore permitted the accurate determination of the stress corresponding to a creep rate of $1 \times 10^{-5}$ per day. This is a criterion adopted and standardised for such materials by the National Physical Laboratory.

The tubular resistance furnaces surrounding the specimens were provided with means for independent adjustment of the current supplied to the upper and lower parts of windings, and by this means temperature gradients along the length of the specimen were substantially eliminated. Delicate thermostats permitted control of the temperature of the specimen to within very close limits, the total change over long periods being of the order of 0·5° C. at a temperature of 400° C. Three individually calibrated thermo-couples were attached to each specimen, and temperature readings were taken daily by means of an accurate potentiometer.

TABLE I. TENSILE TESTS AT ORDINARY AND ELEVATED TEMPERATURES.

| Percentage Composition. | | | | | At Room Temperature | | | At 250° C. | | At 400° C. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Copper | Aluminium (by difference) | Iron | Nickel | Condition (See notes below). | Tensile Strength tons/sq. in. | Elongation % on $4\sqrt{\text{area}}$ | Reduction of Area % | Tensile Strength tons/sq. in. | Elongation % on $4\sqrt{\text{area}}$ | Tensile Strength tons/sq. in. | Elongation % on $4\sqrt{\text{area}}$ | Reduction of area % |
| 97·93 | 2·06 | 0·01 | — | Cold drawn 50% .. .. | 29·0 | 18 | 72 | — | — | 11·9* | 28* | 40* |
| | | | | Annealed 1 hr. 575° C... | 17·3 | 65 | 82 | — | — | 9·1 | 35 | 34 |
| 94·33 | 5·66 | 0·008 | — | Cold drawn 50% .. .. | 46·4 | 20 | 66 | — | — | 9·4 | 33 | 29 |
| | | | | Annealed 1 hr. 575° C... | 27·3 | 81 | 80 | — | — | 9·4 | 21 | 20 |
| 93·11 | 6·89 | 0·002 | — | Sand Cast .. .. .. | 29·9 | 25 | — | 27·7 | 38 | 13·6 | 50 | — |
| 93·20 | 6·76 | 0·04 | — | Extruded .. .. .. | 37·1 | 31 | 38 | — | — | 15·7 | 47 | 43 |
| | | | | Normalised .. .. .. | 34·2 | 32 | 29 | — | — | 13·9 | 44 | 36 |
| | | | | Stabilised .. .. .. | 27·4 | 14 | 11 | — | — | 15·3 | 35 | 34 |
| 88·89 | 11·10 | 0·006 | — | Extruded .. .. .. | 39·7 | 9 | 12 | — | — | 12·4 | 104 | 95 |
| | | | | Normalised .. .. .. | 33·0 | 6 | 11 | — | — | 12·2 | 89 | 96 |
| | | | | Stabilised .. .. .. | 32·6 | 2 | 2 | — | — | 19·7 | 49 | 34 |
| 87·92 | 12·06 | 0·02 | — | Extruded .. .. .. | 35·6 | 7 | 13 | — | — | 16·4 | 49 | 52 |
| | | | | Normalised .. .. .. | 31·1 | 5 | 11 | — | — | 17·2* | 28* | 65* |
| | | | | Stabilised .. .. .. | 44·0 | Nil | 2 | — | — | 22·5 | 21 | 25 |
| 89·23 | 7·92 | 2·85 | — | Sand cast .. .. .. | 33·6 | 34 | — | 26·4 | 38 | 14·1 | 22 | — |
| 87·07 | 10·13† | 2·80 | — | Extruded .. .. .. | 46·9 | 17 | 26 | — | — | 19·4 | 51 | 46 |
| | | | | Normalised .. .. .. | 42·7 | 26 | 25 | — | — | 16·8 | 77 | 59 |
| | | | | Stabilised .. .. .. | 38·6 | 14 | 28 | — | — | 20·3 | 57 | 59 |
| 85·97 | 9·96 | 4·07 | — | Hot forged .. .. .. | 39·7 | 32 | — | — | — | 16·1 | 73 | 62 |
| | | | | Stabilised .. .. .. | 41·0 | 22 | 20 | — | — | 21·6 | 60 | 51 |
| 88·67 | 10·09 | 0·20 | 1·04 | Hot forged .. .. .. | 32·0 | 10 | — | — | — | 20·6 | 48 | 59 |
| | | | | Stabilised .. .. .. | 38·4 | 10 | 10 | — | — | — | — | — |
| 87·80 | 9·69 | 1·10 | 1·50 | Hot forged .. .. .. | 43·0 | 26 | 36 | — | — | 20·8 | 48 | 52 |
| | | | | Stabilised .. .. .. | 36·9 | 28 | 25 | — | — | 20·7 | 41 | 38 |
| 79·03 | 9·63 | 5·63 | 5·71 | Sand cast .. .. .. | 39·4‡ | 8‡ | — | 30·1 | 8 | 22·5 | 7 | — |
| 80·76 | 9·55 | 4·70 | 4·97 | Hot forged .. .. .. | 52·6 | 16 | — | — | — | 24·3 | 37 | 32 |
| | | | | Stabilised .. .. .. | 48·3 | 24 | 33 | — | — | 18·2 | 02 | 67 |

* Results variable due to partial annealing or change of structure during test. Mean values for several tests are recorded.
† Aluminium determined chemically.
‡ These figures fail to meet the requirements of B.S. 1073 for sand cast material of this composition.

NOTES ON COLUMN HEADED "CONDITION."
"Cold drawn 50%" means cold drawn to a reduction in cross sectional area of 50% without annealing.
"Annealed 1 hr. 575° C." refers to the annealing treatment applied to material cold drawn as described above.
The "Sand Cast" test bars were of the form shown in Fig. 4. Green sand was used.
The "Extruded" bars were tested as received, back and front ends being used indiscriminately.
The "Normalised" bars were heated for 1 hr. at 850° C., or in some cases at 825° C., and rapidly cooled in air. Matrix $\alpha + \beta$.
The "Stabilised" bars were normalised as above, reheated for 3 days at 500° C., and slowly cooled during 3 days. Matrix $\alpha + (\alpha + \gamma_2)$.

## Tensile Tests

Table I gives the results of tensile tests at room temperature and at 400° C. on 13 aluminium bronzes of different compositions. Tests at 250° C. are also included in the case of three of the materials in the sand cast condition.

The two alloys containing respectively 2·06% and 5·66% of aluminium come within the category of single phase, cold working materials, and were tested after cold drawing to 50% reduction of cross sectional area, and after annealing the drawn rod for 1 hour at 575° C. In each case the tensile strength at 400° C., after annealing, was a little over 9 tons/sq. in., and the extra strength imparted by the cold drawing operation was not retained under test at this temperature.

The properties of the remaining eleven alloys, which all contained sufficient aluminium to give the duplex type of matrix, were investigated in greater detail. Each of the eight wrought materials was tested in the "as received" condition, either extruded or hot forged, and also after the "stabilising" heat treatment already described. The four extruded samples were also tested in the metastable "normalised" condition. The results indicate no lack of strength and ductility at 400° C., even in the case of the "stabilised" samples where the presence of the $\gamma_2$ phase reduces the elongation values at room temperature. For instance, as an extreme case, the stabilised alloy containing 12·06% of aluminium was without ductility at room temperature, but acquired an elongation of 21% when tested at 400° C. Strengths at 400° C. in the "stabilised" condition were in most cases considerably higher than those of the corresponding "normalised" materials. These observations are important, for they indicate that any transformation of $\beta$ into $\alpha + \gamma_2$, which might occur during service at temperatures in the neighbourhood of 400° C. would not be detrimental to the properties at such temperature, though a certain degree of cold shortness would develop on cooling. Such cold shortness would become dangerous only in the case of alloys of higher aluminium content (11% or 12%) than those normally used, for the elongations of the stabilised 10% bronzes ranged from 10 to 28% at room temperature, indicating that the presence of $\gamma_2$ is considerably less detrimental than might be expected.

At 250° C. the transformation of $\beta$ into $\alpha + \gamma_2$ occurs very much more slowly than at 400° C. and for service in this lower temperature range it therefore becomes important to enquire how long the "normalised" properties may be retained. Table II gives the results of tensile and hardness tests at room temperature on "normalised" extruded 10% aluminium bronze, with and without 3% of iron, after holding at 250° C. for periods up to 4 weeks. After the first week there was a distinct increase in the limit of

TABLE II.

CHANGES IN MECHANICAL PROPERTIES OF EXTRUDED ROD AT ROOM TEMPERATURE DUE TO DECOMPOSITION OF $\beta$ INTO $(\alpha + \gamma_2)$

| Particulars | "Normalised" 1 hr. 825° C. cooled in air | "Normalised" and held at 250° C. for :— 1 week | 2 weeks | 3 weeks | 4 weeks | "Stabilised" i.e., "Normalised" and reheated 3 days 500° C., slowly cooled during 3 days |
|---|---|---|---|---|---|---|
| **10% Aluminium Bronze :** | | | | | | |
| Composition :—Al 10·47%, Fe 0·01%, Cu 85·52% | | | | | | |
| Limit of proportionality | tons/sq. in. 1·8 | 2·1 | 2·7 | — | 3·1 | 8·5 |
| Proof Stress (0·01% extension) | 4·5 | 7·6 | 9·1 | — | 8·6 | 11·4 |
| Tensile strength | 32·8 | 31·7 | 35·3 | — | 32·5 | 27·4 |
| Elongation on $4\sqrt{\text{area}}$ % | 25 | 24 | 21 | — | 18 | 14 |
| Young's modulus lb./sq in. $\times 10^6$ | 14·5 | 14·8 | 15·5 | — | 14·9 | 19·4 |
| Vickers diamond pyramid hardness number | 146 | — | — | — | 162 | — |
| Structure (as seen under microscope) | $\alpha + \beta$ | No change | No change | — | Incipient $\beta \rightarrow (\alpha + \gamma_2)$ | $\alpha + (\alpha + \gamma_2)$ |
| **10% Aluminium Bronze With 3% Iron :** | | | | | | |
| Composition :—Al 10·13% Fe 2·80%, Cu 87·07% : | | | | | | |
| Limit of proportionality | tons/sq. in. 1·8 | 4·9 | 4·9 | 4·5 | 5·3 | 16·5 |
| Proof stress (0·01% extension) | 6·0 | 14·4 | 12·8 | 13·3 | 12·8 | 18·8 |
| Tensile strength | 43·8 | 44·8 | 45·3 | 46·0 | 47·5 | 38·6 |
| Elongation on $4\sqrt{\text{area}}$ % | 21 | 18 | 19 | 18 | 20 | 14 |
| Young's modulus lb./sq. ins. $\times 10^6$ | 16·5 | 18·4 | 17·7 | 18·1 | 17·8 | 19·8 |
| Vickers diamond pyramid hardness number | 161 | 170 | 178 | 179 | 189 | 192 |
| Structure (as seen under microscope) | $\alpha + \beta + (\text{Fe})^*$ | No change | No change | Incipient $\beta \rightarrow (\alpha + \gamma_2)$ | Incipient $\beta \rightarrow (\alpha + \gamma_2)$ | $\alpha + (\alpha + \gamma_2) + (\text{Fe})^*$ |

* (Fe) signifies the iron rich constituent.

TABLE III. NOTCHED-BAR TESTS AT ORDINARY AND ELEVATED TEMPERATURES.

| Percentage Composition : | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Copper | 89·89 | | 87·07 | | 87·91 | 88·23 | 87·70 | 80·66 |
| Aluminium | 10·06 | | 10·13 | | 9·95 | 9·10 | 9·33 | 9·33 |
| Iron | 0·039 | | 2·80 | | 4·17 | 0·34 | 0·99 | 5·20 |
| Nickel | — | | — | | — | 1·53 | 1·96 | 48·1 |
| **Condition** | Extruded and Normalised | Extruded and Stabilised | Extruded and Normalised | Extruded and Stabilised | Hot Forged | Hot Forged | Hot Forged | Hot Forged |
| **Notched-Bar Value in ft. lb. at :—** | | | | | | | | |
| Room temperature | 66 | 8 | 27 | 6 | 13 | 7 | 20 | 13 |
| 200° C. | 58 | 10 | 28 | 6 | 15 | 7 | 25 | 16 |
| 250° C. | 59 | 9 | 34 | 7 | 17 | 10 | 17 | 20 |
| 300° C. | 80 | 9 | 36 | 8 | 20 | 20 | 14 | 22 |
| 350° C. | 58 | 9 | 28 | 6 | 19 | 26 | 20 | 18 |
| 400° C. | 30 | 10 | 15 | 9 | 14 | 4 | 13 | 18 |
| 450° C. | 15 | 14 | 8 | 9 | 12 | 14 | 14 | 17 |
| 500° C. | 13 | 16 | 10 | 13 | 14 | 11 | 16 | 14 |
| 550° C. | 19 | 26 | 21 | 19 | 18 | 6 | 11 | 12 |
| 600° C. | 78* | 73* | 46 | 46 | 38 | 24 | 18 | 14 |

* Specimens incompletely broken. In all other cases fracture was complete.
"Normalised" 1 hour 825° C., cooled in air. Matrix : $\alpha + \beta$.
"Stabilised" 1 hour 825° C., cooled in air : reheated 3 days 500° C., cooled in furnace during 3 days. Matrix : $\alpha + (\alpha + \gamma_2)$.
"Hot forged." The individual test results for materials in this condition showed appreciable scatter owing to lack of initial equilibrium.

proportionality, proof stress and hardness with relatively little change in tensile strength and elongation. Not until the third or fourth week, however, could the incipient change of $\beta$ into $\alpha + \gamma_2$ be detected micrographically, and even at the end of the fourth week the properties remained much more similar to those of the original "normalised" material than to those in the fully "stabilised" condition, included for comparison in the final column of the table.

## Notched-bar Tests

From the tensile tests already described the conclusion was drawn that the presence of the $\gamma_2$ phase diminished the ductility to a considerably lesser degree than is popularly supposed. It seemed important further to investigate this finding by means of the notched-bar impact test, which is a better criterion of embrittlement than the tensile elongation.

Each of the results given in Table III is the mean of several individual tests. These were concordant, except in the case of the two alloys containing 1·53 and 1·96% of nickel respectively, which showed considerable scatter, apparently due to uncontrolled changes taking place in the hot forged material at the temperatures of test.

From a comparison of the results for the two extruded alloys in their normalised and stabilised conditions it is evident that stabilisation causes a considerable degree of embrittlement up to about 450° C., at which temperature the notched-bar values become the same for both types of heat treatment. It should be noted that, had the normalised specimens been held at longer periods in the temperature range between about 250° and 450° C. before testing, they would almost certainly have become more brittle, owing to gradual reversion to the stabilised form. There is evidence of this effect in their marked drop in notched-bar value at about 400° C., where the speed of the reaction is sufficient to be manifest in the relatively short period of heating used. At 600° C. all the alloys, irrespective of their original condition, became tough because the $(\alpha + \gamma_2)$ complex is reconverted into $\beta$ at temperatures above the eutectoid horizontal at 565° C. (See Fig. 1).

## Creep Tests

The three materials selected for intensive study under creep conditions were a straight 10% aluminium bronze, a similar material with the addition of 3% of iron and a ternary alloy containing 10% of aluminium with 5% each of iron and nickel. Each of these materials was tested in the sand cast condition, as well as in the form of extruded rod. In the case of the first two alloys the extruded rod was tested at 250° C. after three different initial heat treatments, namely: (1) normalised, (2) normalised and held at 250° C. for four weeks and (3) stabilised. The complex aluminium bronze, whose mechanical properties were less influenced by heat treatment was used in the "as received" condition alone. Since it was clearly futile to investigate material in the normalised state at 400° C., because of the rapid conversion of $\beta$ into $(\alpha + \gamma_2)$, attention was confined to stabilised material in the testing programme at this temperature.

Sufficient data are presented in Tables IV, V and VI, which refer to the three materials respectively, to indicate the full scope of the experimental work and the shape of the individual creep curves obtained. These tables should prove useful to engineers concerned with problems of design. Table VII gives stresses corresponding to definite creep rates of $10^{-5}$, $10^{-4}$ and $10^{-3}$ per day at the 5th and 40th days, derived from semi-logarithmic plots of creep rate against stress.

The improvement in creep properties effected by the stabilising treatment is apparent from this Table, and it is clear that, under creep conditions, the stabilised is preferable to the normalised form, in spite of the fact that ultimate strength, ductility and notched-bar values at room temperature are inferior. It may be inferred that the improved elastic properties conferred by stabilisation are a dominating factor in resistance to creep. Heating the normalised material

TABLE IV.
CREEP TESTS ON STRAIGHT 10% ALUMINIUM BRONZE.

| PERCENTAGE COMPOSITION: | CAST | WROUGHT |
|---|---|---|
| Copper | 89·91 | 89·52 |
| Aluminium | 10·06 | 10·47 (by diff.) |
| Iron | — | 0·01 |

| Condition (See Text) | Temp. of Test, °C. | Initial Stress tons/sq. in. | Duration of Test, Days | Strain per cent. Immediately after loading | Strain per cent. At 5th day | Strain per cent. At 40th day | Strain per cent. At fracture | Strain rate % per day. At 5th day. Or minimum if earlier. | Strain rate % per day. At 40th day. | REMARKS. |
|---|---|---|---|---|---|---|---|---|---|---|
| Sand Cast | 250 | 5 | 15 | 0·091 | 0·136 | — | Unbroken | 0·0035 | — | Rate still diminishing at end of test |
| | | 8·5 | 27 | 0·293 | 0·541 | — | Unbroken | 0·0123 | — | Rate still diminishing at end of test |
| | | 12 | 34 | 0·709 | 1·181 | — | Unbroken | 0·0346 | — | Rate still diminishing at end of test |
| | | 20* | 0·26 | 6·59 | — | — | 11·69 | 9·6 min. | — | Minimum rate approximate only. |
| Normalised $\alpha + \beta$ | 250 | 1 | 26 | 0·020 | 0·027 | 0·035 | Unbroken | 0·00040 | <0·0001 | Rate at 40th day by extrapolation |
| | | 5† | 51 | 0·091 | 0·121 | 0·153 | Unbroken | 0·0020 | 0·00050 | Rate still diminishing slowly at end of test |
| | | 8·5 | 92 | 0·180 | 0·320 | 0·495 | Unbroken | 0·0084 | 0·0032 | Rate still diminishing slowly at end of test |
| | | 20* | 0·014 | 1·147 | — | — | 1·7 | 1·60 min. | — | Minimum rate approximate only. |
| Normalised and Re-heated 4 weeks at 250° C. | 250 | 1 | 38 | 0·016 | 0·021 | 0·030 | Unbroken | 0·00032 | <0·0001 | Rate at 40th day below sensitivity of measurement |
| | | 5† | 40 | 0·094 | 0·116 | 0·143 | Unbroken | 0·0016 | 0·00032 | Rate still diminishing at end of test |
| | | 8·5 | 62 | 0·168 | 0·255 | 0·370 | Unbroken | 0·0063 | 0·0020 | Rate still diminishing at end of test |
| | | 20* | <2 | 0·771 | — | — | 2·3 | 0·44 min. | — | Minimum rate approximate only. |
| Stabilised $\alpha + (\alpha + \gamma_2)$ | 250 | 8 | 40 | — | — | — | Unbroken | — | 0·00080 | Tested at N.P.L. |
| | | 8·5 | 56 | 0·110 | 0·156 | 0·232 | Unbroken | 0·0045 | 0·0010 | Rate still diminishing at end of test |
| | | 10 | 40 | — | — | — | Unbroken | — | 0·0020 | Tested at N.P.L. |
| | | 12 | 59 | 0·187 | 0·294 | 0·598 | Unbroken | 0·0127 | 0·0080* min. | Minimum rate at about 30th day. |
| | | 12 | 40 | — | — | — | Unbroken | 0·012 min. | — | Tested at N.P.L. Minimum rate at 3rd day. Seems questionable |
| | | 20‡ | 22 | 0·451 | 1·85 | — | 9·0 | 0·24 min. | — | Minimum rate at 4th day |
| Stabilised $\alpha + (\alpha + \gamma_2)$ | 400 | 1·5 | 42 | 0·076 | 0·135 | 0·185 | Unbroken | 0·0040 | 0·0011 | Rate probably diminishing at end of test |
| | | 2 | <0 | — | — | — | Unbroken | — | 0·0027 | Tested at N.P.L. |
| | | 3 | 40 | — | — | — | Unbroken | — | 0·011 | Tested at N.P.L. |
| | | 4 | 18 | — | — | — | 21 | 0·34 min. | — | Tested at N.P.L. Minimum rate at 5th day |
| | | 6 | 2 | — | — | — | 45 | 3·90 min. | — | Tested at N.P.L. Minimum rate at 1st day |

* following 8·5 tons/sq. in. † following 1 ton/sq. in. ‡ following 12 tons/sq. in.

TABLE V.

CREEP TESTS ON 10% ALUMINIUM BRONZE CONTAINING 3% OF IRON.

| Percentage Composition: | Cast | Wrought |
|---|---|---|
| Copper | 86·86 | 87·07 |
| Aluminium | 9·7 | 10·14 (by diff.) |
| Iron | 3·4 | 2·80 |

| Condition (See Text) | Temp. of Test, °C. | Initial Stress tons/sq. in. | Duration of Test, days | Strain per cent. | | | | Strain rate % per day | | Remarks. |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Immediately after loading | At 5th day | At 40th day | At fracture | At 5th day, or minimum if earlier | At 40th day, | |
| Sand Cast | 250 | 5 | 15 | 0·086 | 0·121 | — | Unbroken | 0·0019 | — | Rate still diminishing at end of test |
| | | 8·5 | 27 | 0·150 | 0·216 | — | Unbroken | 0·0070 | — | Rate still diminishing at end of test |
| | | 12 | 34 | 0·362 | 0·600 | — | Unbroken | 0·0249 | — | Rate still diminishing at end of test |
| | | 20* | 19 | 2·32 | 6·90 | — | 13·81 | 0·494 | — | Minimum rate at about 5th day |
| Normalised $\alpha + \beta$ | 250 | 1 | 34 | 0·012 | 0·016 | 0·022 | Unbroken | 0·00026 | <0·0001 | Rate at 40th day by extrapolation |
| | | 5† | 40 | 0·073 | 0·093 | 0·121 | Unbroken | 0·0017 | 0·00048 | Rate still diminishing slowly at end of test |
| | | 8·5 | 56 | 0·138 | 0·220 | 0·352 | Unbroken | 0·0063 | 0·0021 | Rate still diminishing slowly at end of test |
| | | 20* | 80 | 1·033 | 3·20 | 11·7 | 21·1 | 0·26 min. | — | Minimum rate at 3rd day |
| Normalised, Reheated 4 weeks at 250° C | 250 | 1 | 34 | 0·008 | 0·011 | 0·016 | Unbroken | 0·00020 | <0·0001 | Rate at 40th day by extrapolation |
| | | 5† | 48 | 0·071 | 0·089 | 0·116 | Unbroken | 0·0015 | 0·00047 | Rate still diminishing slowly at end of test |
| | | 8·5 | 55 | 0·141 | 0·190 | 0·290 | Unbroken | 0·0060 | 0·0018 | Rate still diminishing slowly at end of test |
| | | 20* | 32 | 1·08 | 3·50 | — | Unbroken | 0·21 min. | — | Minimum rate at 3rd day. Test discontinued prematurely |
| Stabilised $\alpha + (\alpha + \gamma_2)$ | 250 | 8 | 56 | 0·108 | 0·149 | 0·184 | Unbroken | 0·0031 | 0·0005 | Rate still diminishing slowly at end of test |
| | | 10 | 56 | 0·137 | 0·228 | 0·302 | Unbroken | 0·0062 | 0·0012 | Rate still diminishing slowly at end of test |
| | | 12 | 56 | 0·177 | 0·289 | 0·448 | Unbroken | 0·0082 | 0·0032 | Rate still diminishing slowly at end of test |
| | | 20‡ | 59 | 0·416 | 1·10 | 10·15 | 23·4 | 0·12 min. | — | Minimum rate at 3rd day |
| Stabilised $\alpha + (\alpha + \gamma_2)$ | 400 | 2·5 | 47 | 0·041 | 0·108 | 0·196 | Unbroken | 0·0065 | 0·0015 | Rate still diminishing at end of test |
| | | 3·5 | 57 | 0·052 | 0·175 | 0·367 | Unbroken | 0·0090 | 0·0049 | Rate still diminishing at end of test |
| | | 4·5 | 56 | 0·074 | 0·375 | 1·84 | Unbroken | 0·0330 | 0·029 min. | Rate increasing rapidly at end of test |

* following 8·5 tons/sq. in. † following 1 ton/sq. in. ‡ following 12 tons/sq. in.

TABLE VI.

CREEP TESTS ON COMPLEX ALUMINIUM BRONZE CONTAINING IRON AND NICKEL

| Percentage Composition: | Cast | Wrought |
|---|---|---|
| Copper | 80·48 | 80·44 |
| Aluminium | 9·5 | 9·86 |
| Iron | 5·4 | 4·97 |
| Nickel | 4·8 (by diff.) | 4·87 |

| Condition (See Text) | Temp. of Test, °C. | Initial Stress tons/sq. in. | Duration of Test, days | Strain per cent. | | | | Strain rate % per day | | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Immediately after loading | At 5th day | At 40th day | At fracture | At 5th day or minimum if earlier | At 40th day | |
| Sand Cast | 250 | 5 | 27 | 0·092 | 0·110 | — | Unbroken | 0·0009 | — | Rate still diminishing at end of test |
| | | 8·5 | 27 | 0·093 | 0·143 | — | Unbroken | 0·0029 | — | Rate still diminishing at end of test |
| | | 12 | 34 | 0·178 | 0·268 | — | Unbroken | 0·0077 | — | Rate still diminishing at end of test |
| | | 20* | 75 | 0·408 | 0·840 | 2·51 | Unbroken | 0·0585 | 0·0444 | Rate still diminishing at end of test |
| Extruded to meet Specification D.T.D. 197 | 250 | 1 | 49 | 0·012 | 0·015 | 0·016 | Unbroken | <0·0001 | <0·0001 | Rate at 5th day below sensitivity of measurement |
| | | 2·5 | 49 | 0·029 | 0·041 | 0·051 | Unbroken | 0·00036 | <0·0001 | Rate at 40th day below sensitivity of measurement |
| | | 4 | 49 | 0·047 | 0·062 | 0·068 | Unbroken | 0·00084 | <0·0001 | Rate at 40th day below sensitivity of measurement |
| | | 6 | 60 | 0·079 | 0·106 | 0·123 | Unbroken | 0·00132 | 0·00020 | Rate still diminishing slowly at end of test |
| | | 9 | 60 | 0·119 | 0·186 | 0·236 | Unbroken | 0·0027 | 0·00076 | Rate still diminishing slowly at end of test |
| | | 14 | 53 | 0·183 | 0·34 | 0·622 | Unbroken | 0·0133 | 0·0062 | Rate still diminishing slowly at end of test |
| | | 20 | 47 | 0·29 | 0·80 | 3·67 | 4·4 | 0·071 | — | Minimum rate at 5th day |
| | 400 | 3 | 30 | 0·047 | 0·188 | 0·43 | Unbroken | 0·0105 | 0·0057 | Rate at 40th day by extrapolation |
| | | 4 | 30 | 0·060 | 0·266 | 0·76 | Unbroken | 0·0198 | 0·013 | Rate at 40th day by extrapolation |
| | | 5 | 30 | 0·076 | 0·620 | 1·93 | Unbroken | 0·0529 | 0·033 | Rate at 40th day by extrapolation |

* following 8·5 tons/sq. in.

for a month at 250° C. before commencing creep tests at that temperature led to results intermediate between those for the normalised and stabilised conditions, owing to incipient formation of $(\alpha + \gamma_2)$ from the $\beta$. If, therefore, the requirements of service are such that the relatively low creep resistance of the normalised material can be tolerated initially, no harm will be done by using it in that condition because it will tend to improve during service rather than to deteriorate.

The creep tests at 250° C. under 20 tons/sq. in. were carried to destruction, and comparison of Tables IV, V and VI shows that the alloy containing 3% of iron gave consistently longer life and greater extension at fracture than either of the others under these conditions. The break usually went preferentially around the $\alpha$ grains as in Fig. 5, while small cracks, such as that shown in Fig. 6, sometimes appeared in the surface of the specimen not far from the final fracture. No cracking or fracture occurred in any of the specimens tested at 14 tons/sq. in. or less at 250° C.

## Conclusions

For purposes of comparison, a few creep results for tin bronze, gun metals and a copper-silicon-manganese

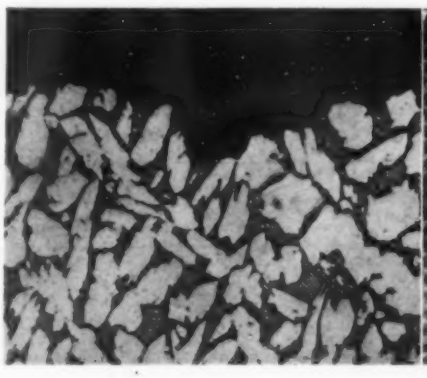

**Fig. 5.—Typical fracture propagated preferentially around the alpha particles. × 100.**

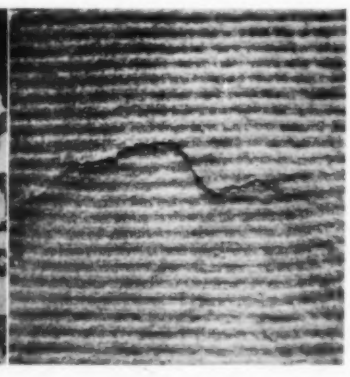

**Fig. 6.—Typical crack in surface of test piece. (The machining marks are perpendicular to the direction of creep). × 20.**

TABLE VII. STRESSES CORRESPONDING TO DEFINITE CREEP RATES.

| Percentage Composition | | | | Condition (see Text) | Temperature of Test °C. | Stresses in tons/sq. in. corresponding with definite creep rates, in strain units per day at the:— | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | 5th day | | | 40th day | | |
| Copper | Alumin'm | Iron | Nickel | | | $10^{-6}$/day | $10^{-5}$/day | $10^{-4}$/day | $10^{-5}$/day | $10^{-4}$/day | $10^{-3}$/day |
| 89·91 | 10·06 | — | — | Sand cast | 250 | <2 | 8·0 | 14·2 | — | — | — |
| 89·52 | 10·47 | 0·01 | — | Normalised, 1 hr. 825° C., cooled in air | 250 | 3·2 | 9·0 | 14·7 | 6·3 | 10·5 | 14·8 |
| 89·52 | 10·47 | 0·01 | — | Normalised and held 4 weeks at 250° C. | 250 | 3·8 | 9·9 | 15·9 | 7·2 | 12·0 | 16·8 |
| 89·52 | 10·47 | 0·01 | — | Stabilised, 3 days 500° C., slowly cooled | 250 | 4·4 | 11·1 | 17·7 | 8·5 | 13·3 | 18·2 |
| 89·52 | 10·47 | 0·01 | — | Stabilised, 3 days 500° C., slowly cooled | 400 | — | c. 2·1 | — | 1·4 | 2·7 | 3·9 |
| 86·86 | 9·7 | 3·4 | — | Sand cast | 250 | 3·5 | 9·5 | 16·8 | — | — | — |
| 87·07 | 10·13 | 2·8 | — | Normalised, 1 hr. 825°C., cooled in air | 250 | 3·8 | 9·7 | 17·1 | 6·7 | 12·3 | 18·2 |
| 87·07 | 10·13 | 2·8 | — | Normalised and held 4 weeks at 250° C. | 250 | 4·2 | 10·1 | 17·0 | 7·0 | 12·6 | 18·3 |
| 87·07 | 10·13 | 2·8 | — | Stabilised, 3 days 500° C., slowly cooled | 250 | 4·7 | 12·0 | 19·3 | 9·5 | 14·5 | 19·5 |
| 87·07 | 10·13 | 2·8 | — | Stabilised, 3 days 500° C., slowly cooled | 400 | — | 3·6 | — | 2·3 | 3·9 | 5·5 |
| 80·48 | 9·5 | 5·4 | 4·8 | Sand cast | 250 | 5·3 | 12·8 | c. 23 | — | — | — |
| 80·44 | 9·86 | 4·97 | 4·87 | Extruded | 250 | 5·1 | 13·1 | 21·2 | 9·8 | 15·3 | 20·7 |
| 80·44 | 9·86 | 4·97 | 4·87 | Extruded | 400 | — | 5·0 | — | — | 3·7 | — |
| Copper | Tin | Zinc | Phos-phorus | | | | | | | | |
| 89·92 | 9·90 | — | 0·03 | Sand cast | 250 | 3·8 | 6·5 | 8·4 | 3·6 | 5·8 | — |
| 88·02 | 9·92 | 2·05 | 0·01 | Sand cast | 250 | 3·3 | 6·4 | 8·4 | 4·0 | 6·3 | 8·5 |
| 87·95 | 8·33 | 3·72 | 0·004 | Sand cast | 250 | 3·5 | 6·9 | 8·7 | 4·6 | — | — |
| Copper | Silicon | Mang. | Iron | | | | | | | | |
| 96·00 | 3·02 | 0·96 | 0·025 | Cold drawn 37% and annealed 1 hr. 575°C. | 250 | — | — | — | 7·1 | 9·4 | — |
| 96·00 | 3·02 | 0·96 | 0·025 | Cold drawn 37% and annealed 1 hr. 575°C. | 400 | — | — | — | 3·8 | 5·4 | 5·9 |

alloy have beeen included in Table VII, the testing technique being the same as that described for the aluminium bronzes. It will be seen that the tin-bearing alloys were much inferior to the aluminium bronzes in their resistance to creep and, while the silicon alloy appeared to be slightly superior at 400° C., this material was handicapped by relatively large initial extensions. In reviewing the published creep data of non-ferrous metals and alloys, Baker[4] found no copper-base material with properties superior to those obtained in the course of the present work for the aluminium bronzes.

For such reasons, and because of their great resistance to oxidation and scaling, the aluminium bronzes, as a class, appear to be the most promising of the copper-base alloys for service at moderately elevated temperatures.

While they cannot, of course, compare with the heat resisting steels and alloys on nickel and chromium, systematic research on the aluminium bronzes would doubtless lead to the development of alloys with even better properties than those recorded in the present paper, which would find a wide variety of applications within the limited temperature range for which they are suitable.

4 B.N.F.M.R.A. published report R.R.A. 449, June, 1937: "Creep of Non-Ferrous Metals and Alloys: A Review of Published Information." By W. A. Baker.


### Acknowledgments

This work formed part of a programme of research on the high temperature properties of copper-base alloys carried out by the British Non-Ferrous Metals Research Association, to whose Director and Council the author is indebted for permission to publish this paper. He acknowledges assistance from past and present members of the Association's Staff.


# The History and Present Status of Emission Spectroscopy as Applied to Industry—II


By John Convey, F.Inst.P.


### Prismatic Spectrographs

THE spectroscopic set-up used by Newton consisted of a slit, a glass prism and a single lens to focus the image of the spectrum on to a screen. Fraunhofer replaced the lens and screen arrangement with a telescope which provided for the actual observation and angular determination of the positions of spectral lines. Babinet overcame the inherent astigmatic error associated with Fraunhofer's apparatus with the introduction of a collimating lens between the slit and the prism. The slit was located in the focal plane of the collimator lens, thus all rays of light after emergence from the collimator were incident on the glass prism as a parallel beam. Kirchoff and Bunsen used both a collimator and telescope. This provided the basic form of spectroscope that was adopted for many years.

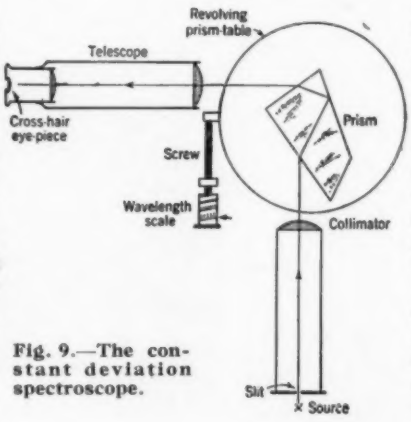


**Fig. 9.—The constant deviation spectroscope.**

In 1904, Twyman, who holds an esteemed position in applied spectroscopy, recognised the need for more convenient and labour-saving spectroscopes and designed the well-known constant deviation spectrometer as illustrated in Fig. 9.

The collimator and telescope are fixed and the prism is rotated by a micrometer screw on the large head of which are engraved the wavelengths of the spectral lines under investigation.

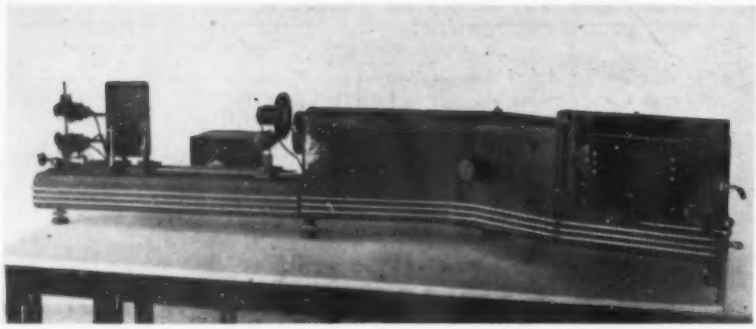

**Fig. 10.—The Bausch and Lomb medium spectrograph.**

The work of De Gramont and Hartley emphasised the need for a good photographic instrument, and in 1909 Twyman developed a fixed adjustment medium quartz spectrograph whose type is illustrated in Fig. 10.

The medium instrument was followed in 1912 by a large quartz spectrograph which has about three times the dispersion of the medium quartz spectrograph. With the establishment of the spectrograph in industrial laboratories the need arose for an instrument whose adjustment would be automatic and labour saving, at the same time capable of operation by unskilled analysts. Semi-automatic instruments were produced, and about 1935 Twyman produced a completely automatic large quartz spectrograph as illustrated in Fig. 11.

## Grating Spectrographs

To-day, the diffraction grating spectrograph which was originally invented by Fraunhofer and later improved by Rowland and Michelson appears to be replacing the prismatic spectrographs in industrial laboratories, especially in America. The Linear dispersion of grating instruments is an asset that is not possessed by prism spectrographs. Modern grating spectrographs differ solely in their type of mounting and various mechanical adjustments. The early optical gratings were ruled on speculum metal which is now replaced to a large extent with aluminium, whose light reflectivity is higher in the ultra-violet than that of speculum. Wood, of John Hopkin's University used glass blanks whose surface is coated with aluminium by evaporation for the production of his gratings. The ruling of the gratings appears to be no longer an art, as several scientific instrument firms in the U.S.A. possess their own ruling machines. Excellent replica gratings from good metal on glass gratings are now available at a fairly low cost. Due to their lower cost and linear spectral dispersion it is probable that in industry, grating spectrographs will find an increasingly wider use. Fig. 12 shows one of the latest types of grating spectrograph produced by the A.R.L.- Dietert Company of America.

This two-meter grating spectrograph is supplied with two gratings of 36,600 and 24,400 lines per inch with respective dispersions of 3·4 and 5·2 angstroms per millimetre in the first order spectrum. Either grating may be brought into use by a small angular shift of the incident beam from one grating to another. A movable 24 in. camera provides for the photography of the spectral region 1,850 to 9,200 angstroms on 35 mm. spectrum film.

Fig. 13 illustrates a grating produced by the Jarrel & Ash Co., of America. This instrument is of the Wadsworth mount type and is fully automatic and stigmatic, with a linear dispersion of 5 amm. in the first order spectrum.

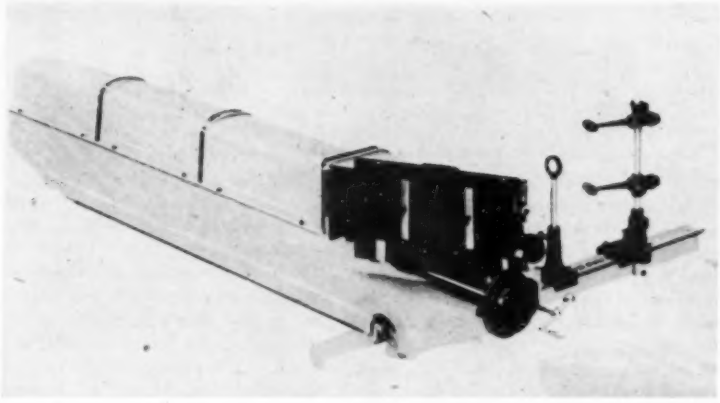

**Fig. 11.—The fully automatic large quartz Hilger spectrograph.**

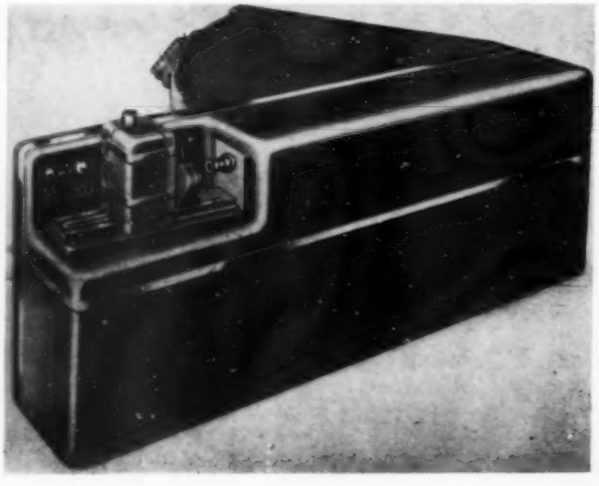

**Fig. 12.—The two meter A.R.L.-Dietert grating spectrograph.**

## The Development of Controlled Light Sources

The story of industrial spectrography from 1935 to 1945 reveals the urgent need for the control of spectral excitation and the carefully planned

investigations directed towards a reduction of this light-source error. The recognition of the characteristic flame spectra of several elements laid the foundation of spectrochemistry. However, except for the alkali metals and a very few others, the low temperature flame of the Bunsen or Meker burners is not a satisfactory method of atomic excitation as very few spectral lines are emitted. An improved flame source was developed by Lundegardh,[26] whereby a solution of the sample under

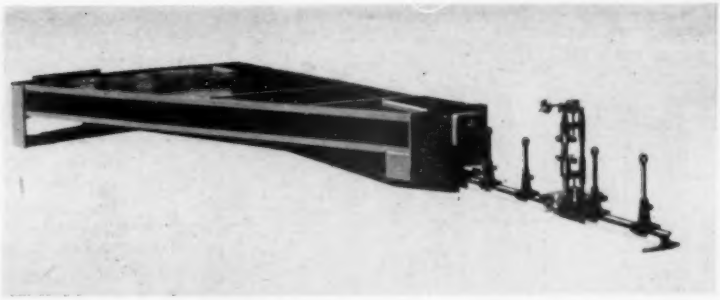

**Fig. 13.—The large Jaco-Wadsworth fully automatic grating spectrograph**

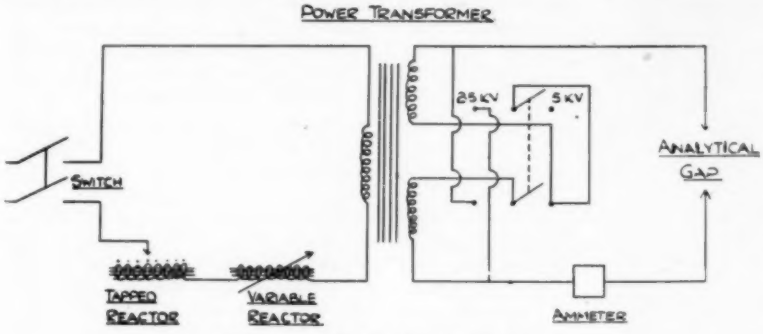


**Fig. 14.—High voltage A.C. arc.**

analysis is fed through an atomiser connected to the air inlet of an acetylene or oxyacetylene flame. Although the spectra are relatively poor in the number of lines emitted some thirty-two elements may be determined with this method. Many modifications of Lundegardh's apparatus have been produced in recent times particularly those of Ramage[27] and Mitchell.[28] A good comprehensive survey of spectrochemical analysis using flame methods of spectral excitation has been published by Cholak and Hubbard.[29]

As many of the samples received for analysis in industrial laboratories are of the solid conducting type, the use of an arc discharge for spectral excitation was soon recognised and it soon became the popular method. The direct current arc between two solid electrodes is a simple source unit which possesses a high sensitivity for the detection of traces of impurities in a sample. Unfortunately, this source is subject to erratic fluctuations that limit it to work of a semi-quantitative or qualitative role. Wandering of the discharge produces variations in its temperature, volume, and density, with the selective excitation of elements of low boiling point that enter the discharge before the higher melting point elements. Experience showed that some of the variations would smooth themselves out over a whole exposure and the process could be improved in several cases by superimposing exposures. Many attempts have been made to stabilise the direct current arc. The use of reactance in the circuit produces steadiness, although excessive reactance reduces the sensitivity of the arc. Rotation of the electrodes has also helped.[30, 31] Electrodes of various geometrical designs[32, 33] producing an extremely rapid volatilisation of the sample were found to increase the absolute spectral intensity from two to five times as intense as that obtained with the normal sample graphite rod arc employing the same current. In addition, the arc was very markedly stabilised and thus enhanced the reproducibility of analysis.

Gerlach[34] introduced the interrupted d.c. arc for the analysis of low melting alloys, whereby the electrodes are repeatedly brought into contact and separated. Pfeilsticker[35] improved the interrupted arc method without any movement of the electrodes. A simple 200 volt a.c. arc is triggered at the peak of each cycle with a Tesla discharge. Modified forms of this circuit are employed in several laboratories.

26 Lundegardh, H. *Die Quan. Spek. d Elemen t.* Jena. Gustav Fischer, I, 1929. II, 1934.
27 Ramage. *Nature*, 123, 601. (1929).
28 Mitchell, J. *J. Soc. Chem. Ind.*, 4, 95. (1941).

29 Cholak, J., and Hubbard, D. M. *Ind. Eng. Chem.* (Anal. Edit.), 16, 12, 728–734. (1944).

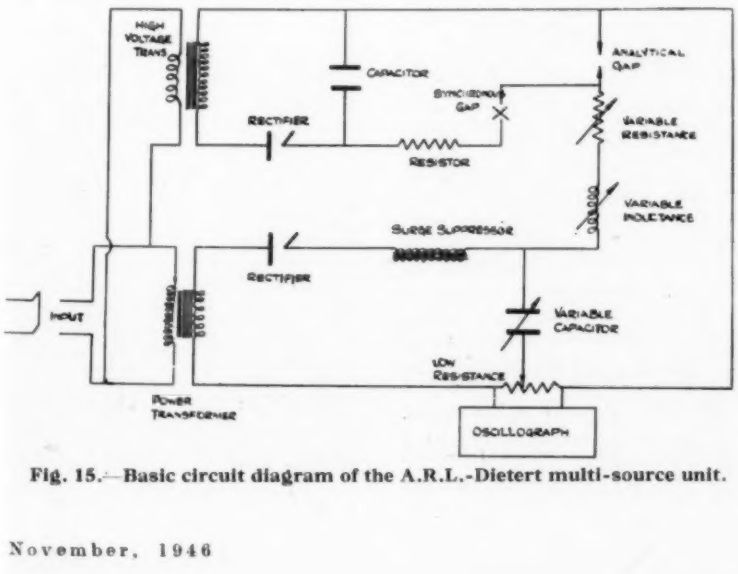


**Fig. 15.—Basic circuit diagram of the A.R.L.-Dietert multi-source unit.**

30 Vincent, H. B., and Sawyer, R. A. *J. Appl. Phys.*, 8, 3, 163–174. (1937).
31 Taylor, R. *J. Sci. Insts.*, 19, 1, 11. (1942).
32 Hasler, M. F. *J. Opt. Soc. Am.*, 31, 1, 21. (1944).
33 Churchill, J. R. *Ind. Eng. Chem.* (Anal. Edit.), 17, 2, 66. (1945).
34 Gerlach, W. *Clinical and Path. Appl. of Spect. Anal.* (1934). A. Hilger, Ltd., London.
35 Pfeilsticker, K. *Z.f. Electrochemie*, 43, 8, 719. (1937).

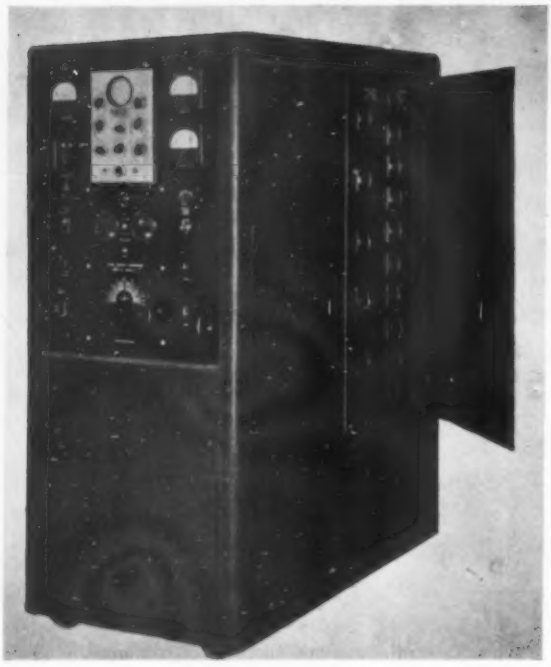

**Fig. 16.—The complete A.R.L.-Dietert multi-source unit.**

Duffendack and Wolfe[36] produced a high voltage a.c. arc source which consists of a high voltage transformer which steps up the current from 120 to 2,200 peak volts with a power rating of 5 kva. A commercially available source has been produced by the A.R.L.-Dietert Company of America. Fig. 14 represents a schematic diagram of the main circuit.

A variable reactor is employed in the 200–300 volts primary circuit of the power transformer. The secondary circuit of the transformer is designed to deliver 2,500 or 5,000 peak volts on no load and is directly connected across the arc. Change of reactance allows the current in the arc to be varied continuously between 1 to 5 amps in the 2,500 volt, and 1 to 2½ amps in the 5,000 volt range. This arc unit has been extensively used in recent years.

For analysis of high quantitative accuracy it was found that a condensed spark source was required. Generally it consists of a high voltage transformer, providing from 10 to 40 kilovolts in the secondary, a condenser that is charged once every half cycle by that high voltage and a discharge circuit connected across the condenser and consisting of inductance, resistance, and one or more spark gaps in series. Early studies of this source indicated that its stability was affected by non-reproduction of the breakdown potential of the analysis gap. To overcome this defect Feussner[37] produced a controlled condensed spark circuit wherein a rotary interrupter inserted in the secondary unit allows the discharge to take place only when the condenser is at its peak voltage. Vincent and Sawyer[38] simplified the Feussner unit. This type of source is employed in most of the iron and steel laboratories of America. The A.R.L.-Dietert Company have produced a high voltage spark unit similar to that of the Vincent and Sawyer network.

To extend the range of analysis to concentrations in ferrous analysis, Dietert found that ultra-violet irradiation of the rotary gap was beneficial. Simpson[39] found this irradiation of the interrupter gap improved the reproducibility of analyses of high steel constituents by 40%.

Levy[40] controls the simple condensed spark discharge by making the charging and discharge parts of the cycle completely independent. This is maintained by short circuiting the analysis gap with a coil of low inductance during the charging period of the condenser. Malpica and Berry[41] produced an electronic spark generator. This scheme provides a stable discharge obtained by controlled charging of the condenser. In 1943 Hasler and Dietert[42] developed and produced commercially a spectral excitation unit which can give a wide variety of excitation conditions ranging from spark to arc charactertistic discharges by suitable variations of the inductance, capacitance, and resistance factors. This source is known as the multi-source unit.

Fig. 15 illustrates the basic circuit diagram which is comprised of a high voltage ignitor and a low voltage rectifier set-up to charge the main condenser bank. Fig. 16 shows the completed unit.

In this unit the complete separation of the charge and discharge period makes each independently controllable. Applications of this circuit have provided notable improvements of many spectrochemical procedures as witnessed by the author in United States laboratories. In addition, the controlled behaviour available in this unit has given a great impetus to the study of electrical discharges and direct reading methods of spectral intensities. Similar source units are under investigation in several laboratories. It may be concluded that the past two years of research into spectral excitation has presented industrial spectroscopy with a single unit that replaces the older assortment of d.c. arc, a.c. arc, and high voltage spark units. Furthermore, the spectrographic analysis of high alloy contents is rapidly becoming an established procedure when multi-source units are employed. Incorporated in the Dietert source units are the exposure, timing, safety circuits, meters, etc., which render the exposure of a sample completely impersonal.

*To be continued.*

36 Duffendack, O. S., and Wolfe, R. A. *Phys. Rev.*, 40, 1038. (1932).

37 Feussner, O. Eisenhutten, 40, 6, 551. (1933).
38 Vincent, H. B., and Sawyer, R. A. *J. Appl. Phys.*, 8, 3, 163. (1937).
39 Simpson, S. E. *J. Opt. Soc. Am.* 35, 1, 40. (1945).
40 Levy, J. *J. Opt. Soc. Am.*, 35, 3, 221. (1945).
41 Malpica, J. M., and Berry, T. M. *Gen. Elect. Rev.*, 9, 563. (1941).
42 Hasler, M. F., and Dietert, H. M. *J. Opt. Soc. Am.*, 33, 4, 218. (1943).

# Co-Deposition of Tungsten and Nickel

By L. E. Vaaler and M. L. Holt

A NEW aqueous plating bath for the electro deposition of tungsten nickel alloys is described. The bath contains sodium tungstate, citric acid, nickel sulphate, and ammonium hydroxide. When this type of bath was operated at various bath concentrations and conditions of electrolysis, it was found to yield cathode deposits containing from 10 to 35% tungsten. The maximum tungsten content was obtained from a bath of low nickel concentration, high tungstate concentration, minimum citric acid concentration, a bath pH of about 7, a high bath temperature, and a fairly high cathode current density. The most satisfactory bath contained 20 g./L $NiSO_4.6H_2O$; 50 g./L $Na_2WO_4.2H_2O$, 66 g./L citric acid, and a definite amount of $NH_4OH$ to give a bath pH of about 8.

From *Electrochem. Soc.*, preprint 90-99.

# Magnesium Casting Alloys——— Their Production and Use

By G. B. Partridge

*Many magnesium alloys have been developed, but about half a dozen now find general application, and these have been developed since 1920. During the war, much progress was made in improving and standardising the quality of these alloys, but the same type of alloys continue to be used as was used before the war. Magnesium is one of the few engineering constructional metals in this country which is, and can be, wholly self-supporting, and there is a very great field for increased applications for its alloys, based on their wider and more imaginative use. To make fuller use of these alloys there is a need for closer collaboration between the specialist, the designer and user, and this article is the first of a series designed to lead to a better understanding of their production and use.*

THE full story of how magnesium and its alloys were first made in this country and their growth and progress has been admirably told elsewhere,[1] and readers who are interested in the early trials and general history of the actual extraction of the metal from its ores are recommended to read the article referred to. A brief review, however, will enable a proper perspective to be taken of the position.

Prior to 1936 all magnesium used in this country was imported from Germany and America. In that year, however, works were erected at Clifton Junction, near Manchester to extract the metal from its ores. They were built under the direction of Magnesium Elektron, Ltd., under patents purchased from Germany. This plant designed for a capacity of 1,500 tons per annum produced its first metal late in 1936. Shortly afterwards the Air Ministry instructed Magnesium Elektron, Ltd., as well as other producers entering the field at that time, to extend its plant by 2,500 tons annual capacity. Just before the war production at the Clifton Junction works was about 3,000 tons per annum, but production greatly increased during the war and with other extraction plants rose to about 33,000 tons per annum. So vital was this metal to the needs of the Air Ministry that its use was strictly controlled. Even this drastic step was not sufficient, and a serious shortage occurred which severely taxed producers until additional extraction plants were made available to ease the position.

The ending of the war brought about a radical change, and the demand for magnesium dropped considerably. The reasons for this temporary setback are twofold, one being that the cessation of hostilities caused an unavoidable dislocation of industrial equilibrium and the second the fact that the material had been denied to many potential consumers for a period of approximately six years. This fact is important, and it should be realised that, because of its vital contribution towards winning the war, magnesium has suffered a very serious reverse. Its industrial history is very short, but enormous strides in its technical progress have been made which can be regarded as a tribute to the industry's vitality.

Now that there are no restrictions on the use of the metal, it can be applied to replace or supplement other metals in short supply, to improve products, or, entirely on its own properties, it may be applied to a wide range of peace-time industrial products. As a result of its usefulness in the aircraft and allied industries, it is obvious that a great field for increased magnesium alloy applications exists. To make the fullest use of these alloys, however, involves closer collaboration between the specialist, the designer and the user. This collaboration is advisable in the early design stage, so that the advantages of these alloys may be exploited. In order to assist to a better understanding of the capabilities of these alloys, various aspects of the subject will be discussed with a view to facilitating in true perspective the fundamental properties of the alloys and guidance in their use.

## Modern Extraction Process

The dark clouds of war made those responsible for magnesium production, think in terms of national self-sufficiency, and thanks to their energy and resources to-day it is the only metal used on a large scale in engineering and general industry which is completely independent of imports of any kind.

As this discussion is primarily concerned with the position of magnesium and its alloys to-day, it is not necessary to delve into the actual history of extraction processes, but a brief outline of the up-to-date process will be of interest.

Sea water contains a small amount of the magnesium salt, magnesium chloride, and modern magnesium is produced by the reaction of sea water with dolomite limestone which contains magnesium as magnesium carbonate, the resultant metal being derived partly from the sea and partly from the limestone supplies which are abundant in this country. The dolomite which is a double carbonate of magnesium and calcium is heated and then slaked with water to produce calcium and magnesium hydroxide with some magnesium oxide which is now brought into reaction with the magnesium chloride present in sea water. The final product is magnesium oxide, and the equations showing the process are as follows :—

$$\underset{\substack{MgCO_3\\(\text{Dolomite})}}{CaCO_3} + \text{Heat} \longrightarrow \underset{\text{slaked}}{CaO,\ MgO}$$

$$\downarrow$$

$$Ca(OH)_2\left[\begin{matrix}MgO\\Mg(OH)_2\end{matrix}\right]$$

$$\underset{(\text{Sea water})}{MgCl_2} + Ca(OH)_2\left[\begin{matrix}Mg(OH)_2\\MgO\end{matrix}\right] \longrightarrow \underset{\text{Heated}}{2Mg(OH)_2} + CaCl_2$$

$$\downarrow$$

$$2MgO + 2H_2O$$

It is clearly seen that the above process which is operated by British Periclase, Ltd., and The Washington Chemical Co. is only dependent upon English raw materials.

The next step in the process is the formation of anhydrous magnesium chloride from the oxide produced above, and this is the starting point at the works of Magnesium Elektron, Ltd., where the incoming magnesium oxide is finely ground, mixed with coal and small amounts of other substances, wetted with magnesium chloride solution and then calcined.

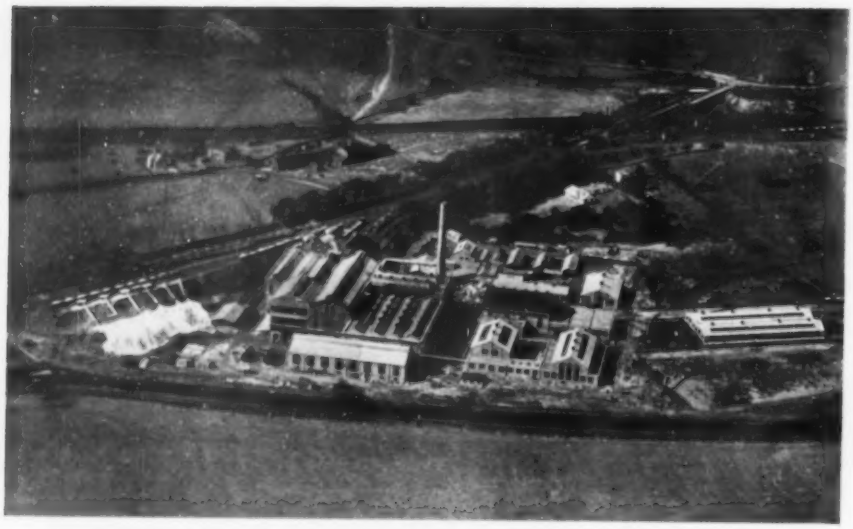

**Fig. 1.—The Clifton Junction plant of Magnesium Elektron, Ltd.**

The calcined material must be very porous for the next operation is chlorination, and unless the chlorine is brought into intimate contact with the whole, much material would be wasted with consequent uneconomic working. Formerly, to obtain this necessary porosity, peat was added to the mixture, and a lengthy process resorted to, but, to-day, mainly after concentration on the mechanical aspects, considerable simplification has taken place with more efficient working. Chlorine is passed over the MgO mixture at about 900° C., whereupon magnesium chloride is formed which is tapped off from time to time as a liquid and is immediately taken to the electrolysis plant and added to those cells which require replenishing with electrolyte. Electrolysis of the $MgCl_2$ is going on continuously and molten magnesium is formed as tiny globules which conglomerate until sufficient accumulates to require removal from the cell; this is done by hand ladles chiefly, about every 24 hours. This crude electrolytic material is of about 99·8% purity and is usually in 56-lb. cones which are contaminated with salts from the electrolytic cell.

Refining of this product is carried out in large 2-ton capacity steel pots where fluxing takes place to remove the impurities after which the superheating and rapid cooling cycle is carried out and the metal tapped out as ingots weighing about 5 lb. each, which is a convenient size for subsequent handling. This constitutes pure magnesium ingot for which there are many uses in industry, but the material as it stands is of little use for engineering applications so the bulk of the output is turned out as "Elektron" alloy, and this is done by the addition of the necessary elements during the fluxing stage in the 2-ton pot. In considering the future, a word must be added about electricity which is vital to the production of the metal. Cheaper electricity means cheaper magnesium, and at the moment there is a continual fight against rising costs especially the cost of coal, it appears probable, therefore, that such hydro-electric schemes as the Severn Barrage would be of immense value to the magnesium industry in the future.

There is a variation of the above process at the Dow Company in America, in which the MgO after its extraction from sea water is converted into magnesium chloride with HCL. This salt is crystallised and carefully dehydrated, whereupon the metal is then extracted by electrolysis in the normal manner.

Two other processes are worthy of mention, the first a thermal process, in which MgO is reacted with carbon at extremely high temperatures, and the second in which calcined dolomite is reduced with silicon in the form of FeSi, this taking place in vacuum.

## Alloying Elements

Magnesium as a pure metal is of little use in the general engineering industry, but as the basis of the ultralight alloys known as Elektron it is invaluable. Magnesium-base alloys are the lightest commercial metals available to industry to-day, their general specific gravity being 1·84 which is one quarter the weight of iron and steel and two-thirds the weight of aluminium alloy. This fact of lightness must be emphasised as it is an outstanding feature of the whole series of alloys and the potentialities of the material cannot be fully realised by engineers unless they can obtain personal experience of this most important property.

Sometimes disparaging remarks are passed about the material, but they result from lack of knowledge or experience since many have no real idea, from the mere written word, of the vast difference in weight between magnesium alloys and iron and steel. Designers and engineers are urged to get practical knowledge of this relative weight value and so regard the properties of the material in a true light.

The main alloys are of the quaternery type and contain the added elements aluminium, zinc and manganese together with small amounts of impurities. The most important addition is that of aluminium, and the equilibrium diagram of the system Mg-Al is shown in Fig. 2. It is unfortunate that this diagram commences from the aluminium side, but it is developed from the most recent work on the subject by Dr. G. V. Raynor and is published by the Institute of Metals.

The low specific gravity of aluminium

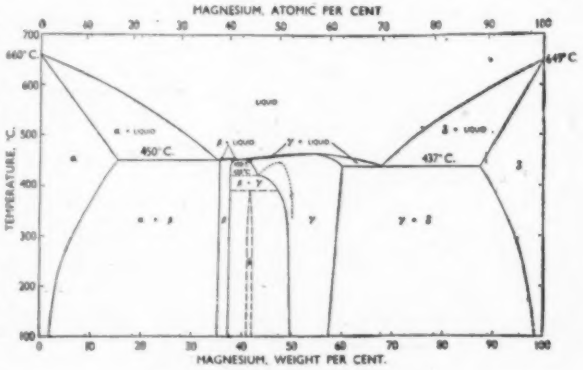


**Fig. 2.—The system Al-Mg.**

and the ease with which it alloys with magnesium make it ideal for the purpose. The useful range of aluminium addition is from 3% to about 10% and as the higher figure is approached the effect is more critical especially with regard to the heat-treatable alloy. Above 11% of aluminium the metal rapidly becomes extremely brittle, possesses a glass-like fracture and is rendered useless, contamination by aluminium alloys in the foundry must therefore be avoided.

Small additions of aluminium form a solid solution referred to as $\alpha$, but as the amount increases the compound $Mg_4Al_3$ appears, the metal becoming increasingly harder; meanwhile the maximum tensile and elongation values in the as-cast state reach a maximum at about 6% aluminium, but, for other reasons which are discussed later, this percentage is rarely used to-day.

Zinc is the next important metal which is used to provide high strength and elongation in magnesium alloys, but as it is necessary to add more zinc to the alloy to obtain the same effect obtained with a smaller amount of aluminium it is not used alone but to supplement the effect of aluminium.

What might be termed the high zinc alloys of magnesium were very common before the war for commercial purposes, but research has tended to prove that such alloys are liable to increased amounts of micro-porosity, and in this country to-day they are completely out of favour. Their use has persisted in the United States, however, as it was thought that the presence of zinc largely influenced the corrosion behaviour of the alloys, but recent work apparently disproves this view. Manganese is added because of the very important part it plays in increasing resistance to corrosion. Iron appears to be the main cause of corrosion in magnesium alloys, and manganese acts as a refiner or precipitant, the process employed being especially effective in the aluminium containing alloys as the presence of aluminium substantially reduces the solubility of manganese in magnesium.[2]

Of the impurities present in the usual alloys, reference has already been made to iron which should be kept as low as possible. It is regularly stated that iron melting units for magnesium are not coated as in aluminium melting because the magnesium does not attack the iron, but this is not correct since during the superheating process relatively large amounts of iron are dissolved in the metal and are precipitated out with falling temperature until at the melting point about 0·02% remains. Some of the latest work on the iron content of magnesium alloys will be discussed in the section dealing with corrosion. Silicon is always present, it being impossible to prevent some particles of sand from getting into the melting pots where they react with the molten magnesium to form $Mg_2Si$. It becomes increasingly insoluble and is clearly seen in the microstructure as bluish cuboids, but sometimes in the form of Chinese script.[3] The risk of corrosion is increased and the alloy tends to lose elongation, the insoluble $Mg_2Si$ particles acting as extra $\beta$ phase. Lead, tin and copper are usually present in small amounts and have little importance except that too much copper is undesirable from the corrosion point of view. The presence of nickel is fatal to the corrosion resistance, and it is imperative to keep the nickel content at an absolute minimum. It is known to-day that even minute quantities of nickel are very detrimental to the corrosion resistance and future work will no doubt bring further enlightenment on this point.

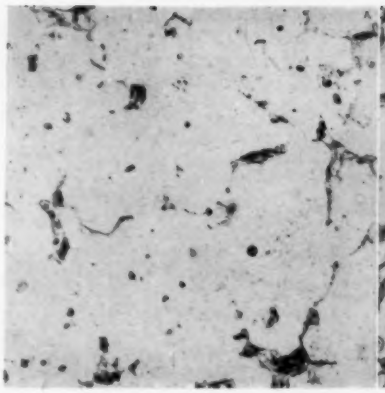

**Fig. 3.—Elektron alloy AZ31. Small amounts of compound are appearing at the grain boundaries ×100.**

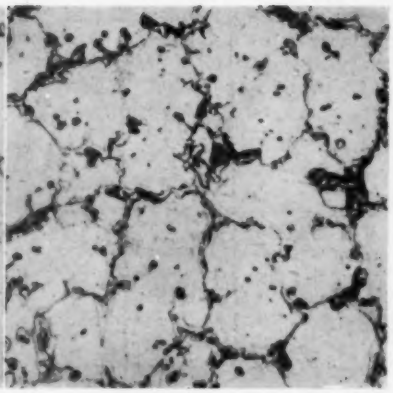

**Fig. 4.—Elektron alloy A8. The network of compound and "eutectoid" is seen in the $\alpha$ matrix ×100.**

## Composition and Properties

The compositions and properties of industrial magnesium alloys, together with the relevant D.T.D. specifications are shown in Table I. The British standards Institution also issue specifications for the Elektron alloys AZ91, A8 and AM503, the numbers of which are from BS 1272-1280, they are nominally the same as the listed D.T.D. specifications. As the main alloys derive their most important physical properties from the amount of aluminium they contain, it will be best to discuss first the low aluminium containing alloy, namely AZ31.

This alloy possesses great elongation in the as-cast state and has a very good tensile strength. Unfortunately, its proof stress is one of the poorest in the Elektron series, and it does not lend itself to rigid applications or to structural parts requiring stiffness. Its main application has been in the manufacture of brake shoes, although the early German technicians were very insistent upon its pressure tightness properties which they said were at their best when the metal was allowed to solidify with a coarse grain structure. As this results in a lowering of mechanical values other alloys are preferred for pressure tightness work in this country. The microstructure is uninteresting and is shown in Fig. 3, the background consisting of solid solution of aluminium in magnesium with the compound $Mg_4Al_3$ just beginning to appear at the grain boundaries, manganese particles and possibly some iron are seen dotted about the general matrix. Other alloys similar to AZ31 but which vary slightly in composition are supplied for purposes requiring extreme ductility.

Before illustrating further alloy structures it will be as well to explain that the alloys do not solidify entirely according to the equilibrium diagram which indicates primary crystallisation of solid solution followed by eutectic. Instead, what is called a "degenerate" eutectic solidifies, which consists of more or less massive particles of $Mg_4Al_3$ associated with its solid solution. Further cooling produces darkly etched patches of very finely divided $Mg_4Al_3$ of lamellar formation, and such effects are known as "eutectoid." This "eutectoid" formation is produced by the breakdown of the supersaturated solid solution, and although the term is not strictly correct it is the easiest way of rapidly describing this

particular phenomenon. The $Mg_4Al_3$ is pure white and it has been proved by varying the type of illumination used with the microscope that all these dark areas are really white and simply different forms of the compound.

Passing reference must be made to the alloy AZG which contained 6% aluminium, 3% zinc, with the usual small quantity of manganese. This alloy was used a great deal before the war for commercial castings and possessed very good mechanical properties in the as-cast condition. Due to the occurrence of micro-porosity in magnesium castings and its detrimental effect upon their physical properties much work was done to try and obviate this defect, and it was found that AZG had a greater tendency[4] towards micro-porosity formation than the other Elektron alloys. Its use was, therefore, discouraged, and to-day the A8 alloy is most commonly used in its place.

The A8 alloy contains about 8% aluminium, 0·5% zinc, 0·2% manganese, and it is usually supplied in one of three forms that is as-cast, annealed or solution heat-treated. It is possible to solution heat-treat and follow with a precipitating treatment as is done for the D.T.D. 285 alloy, but there has not been much call for the material as described, so no specification exists. The A8 alloy is possibly the most important of the Elektron series, many hundreds of thousands of castings having been supplied in this material. Its mechanical values in the as-cast state are quite good, and it is suitable for parts taking moderate shock.

The microstructure is shown in Fig. 4, and against the background of a solid solution is seen the network of $Mg_4Al_3$ with the lamellar eutectoid formation showing at intervals, also in this example particles of the silicon compound $Mg_2Si$ can clearly be seen in Fig. 5.

An interesting development was the introduction of the so-called annealing treatment for castings previously called for in the as-cast condition. The treatment which is the short one of 4–8 hours at 250° C. was intended to act as a casting stress relief anneal and as such is excellent, but the metallurgical considerations are also worth consideration. At this comparatively low temperature the super-saturated solid solution yields up finely divided $Mg_4Al_3$ and the changed structure is shown in Fig. 6, it must be remembered that this is a direct effect following from the structure shown in Fig. 4, not a true precipitation such as follows from solution heat-treatment. Many castings are supplied in this state

**Fig. 5.—A8 at higher magnification, showing the blue particles of $Mg_2Si$. ×500.**

to-day, and it is important to note that there is an increase of Brinell hardness, an improvement in proof stress and tensile strength with only a slight decrease in elongation. The fatigue resistance of the material in this condition is also good, and it is peculiarly suitable for parts required to work at moderately elevated temperatures, that is up to 150° C.

The structure of A8 alloy after solution heat-treatment to spec. D.T.D. 289 is shown in Fig. 7. All the different types of $Mg_4Al_3$ have been dissolved in the matrix leaving the outline of the grain boundaries and the insoluble manganese, iron and $Mg_2Si$ as visible particles deposited throughout. In this condition the material is extremely useful and possesses remarkable resilience and elongation which makes it eminently suitable for a variety of castings demanding high shock resistance—a notable example is the aircraft landing wheel, which to-day is almost exclusively made in this material.

A rise in aluminium content to over 9·0% introduces the AZ91 alloy, which has in certain conditions the highest proof stress of the aluminium containing series. This increase of aluminium content increases the hardness of the material, whilst lowering its tensile strength and elongation slightly in the as-cast condition, this being explained by the increase of excess $Mg_4Al_3$ $\beta$ phase which remains undissolved.

The heat-treatments applicable to AZ91 follow the pattern set for the A8 alloy, and many castings are supplied in the annealed condition. With the exception that the elongation is somewhat lower the same effect is produced metallurgically as with the annealed A8, and the fatigue properties are once again very good. The material is stable and very suitable in applications required to work at temperatures up to 150° C.

Solution heat-treatment is applied exactly as to the A8 alloy, but to specification D.T.D. 281, and the effect is similar except that there is more $\beta$ phase in this alloy, and it is more difficult to take completely into solution with a resultant shortening of physical values. In Fig. 8 is shown the structure of a test bar which gave poor values, the reason for which is easily seen in the pools of undissolved $Mg_4Al_3$. Control of grain size is, therefore, of paramount importance as unless the $\beta$ phase has maximum dispersion it becomes impossible to take it into solution in an economic manner. It is at this stage in the aluminium-containing Elektron series, where the aluminium content becomes critical, as it rises over 9·5% the difficulties of complete solution treatment increase rapidly and the alloy is liable to become somewhat erratic, capable of showing abnormal physical properties, but at other times just managing to pass specification figures.

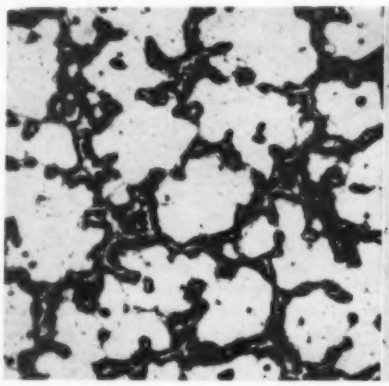

**Fig. 6.—A8 annealed. Fine precipitation of the super-saturated a solid solution. ×100.**

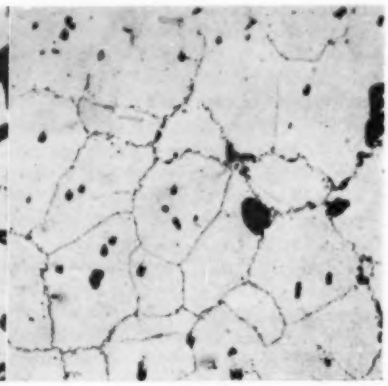

**Fig. 7.—A8 to D.T.D. 289. Solution treatment has completely dissolved all compound. ×100.**

A specification exists for the material in the fully heat-treated condition, namely D.T.D. 285 which consists of a low temperature precipitation after solution heat-treatment. The treatment can apply to A8, although no official specification exists yet the extra elongation as compared with AZ91, which is obtained with fully heat-treated A8, should make it an attractive proposition. The structure of AZ91, after low temperature precipitation, to D.T.D. 285 is shown in Fig. 9, and the masses of dark $Mg_4Al_3$ show the result of this treatment. The alloy realises the highest proof stress of the series from this treatment, but unfortunately it is accompanied by a shortening of the elongation—the material is suitable for applications requiring maximum ridigity.

The high manganese containing alloy known as AM 503 to D.T.D. 140B is important because of its special anti-corrosion and welding properties. It presents some difficulty in the foundry and is generally a poor casting alloy, but as a finished casting it is easily welded and is suitable for fitting to fabricated parts. The corrosion resistance is very good and the alloy is capable of withstanding marine conditions, but it has lower strength than the aluminium containing type of magnesium alloy. Small additions of other elements, for example, aluminium and cerium have been tried to overcome the difficulties met with in casting this alloy but with little success.

There is one other alloy which should be noted and that is Magnuminium 299 which departs from the usual type in containing tin and sometimes silver. It has good shock resistance in the as-cast or annealed conditions, but is not recommended for work at elevated temperatures.

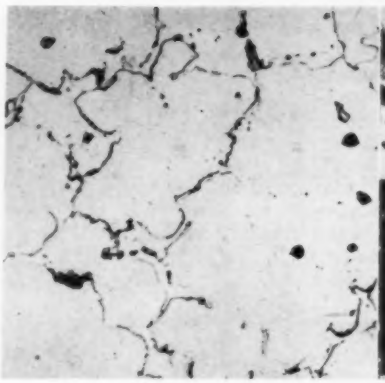

**Fig. 8.—AZ91 test-bar failure. The fairly large grain size has concentrated the $Mg_4Al_3$ into large pools which have resisted solution. ×100.**

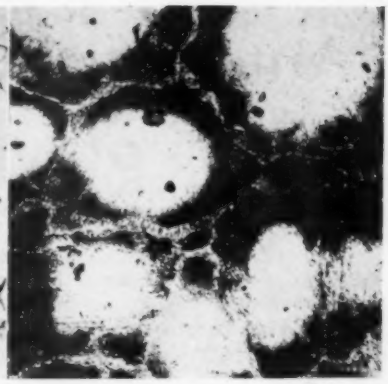

**Fig. 9.—AZ91 solution and precipitation treated. ×100.**

*To be continued.*


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TABLE I.—MAGNESIUM ALLOY, SAND AND GRAVITY DIE CASTINGS

| SPECIFIED | | | | | | | | | | | | | | TYPICAL | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| COMPOSITIONS % | | | | | | | | Heat Treatment | | Mechanical Properties | | | | | | Mechanical Properties | | | | |
| Al | Cu | Mg | Si | Fe | Mn | Ni | Zn | Solution | Precip'n. | 0·1% P.S. | U.T.S. | Eln. | Bn. | Alloy | Specificat'n | 0·1% P.S. | U.T.S. | Eln. | Bn. | Izod |
| **SAND CASTINGS** | | | | | | | | | | | | | | | | | | | | |
| 7·5 | — | R | — | — | 0·15 | — | — | None | None | (4·5) | 9 | 2 | — | A.8 | DTD 59B | 4·5 | 10·0 | 3 | 55 | 1·1 |
| 9·0 | 0·15 | | 0·3 | 0·05 | 0·4 | 0·01 | 1·0 | 4–8 hr. 250° C. | | (4·5) | 9 | 2 | — | A.8 | DTD 59B | 5·0 | 10·0 | 2 | 60 | 0·8 |
| | | | Sn 0·10 | | | | | | | | | | | | | | | | | |
| | | | Cu+Si+Sn+Fe+Ni—0·40 | | | | | 12 hrs. 435° C. | None | (4·5) | 13 | 6 | — | A.8 | DTD 289A | 4·5 | 15·0 | 10 | 55 | 4·0 |
| | | | | | | | | Do. | 8 hrs. 210° C. | (6) | (13) | (2) | — | A.8 | A.8 (S.P.) | 6·0 | 15·0 | 3·5 | 70 | 1·6 |
| 9 | — | R | — | — | 0·15 | — | — | None | None | (4·5) | 8 | — | — | AZ.91 | DTD 136B | 4·5 | 8·5 | 1 | 55 | 0·6 |
| 10·5 | 0·15 | | 0·3 | 0·05 | 0·4 | 0·01 | 1·0 | 4 hrs. 250° C. | | (5·0) | (8) | — | — | AZ.91 | DTD 136B | 5·0 | 9·0 | — | 65 | 0·7 |
| Sn 0·10 | | | Cu+Si+Sn+Fe+Ni—0.40 | | | | | | | | | | | | | | | | | |
| | | | — Do. — | | | | | 16 hrs. 435° C. | None | (5·0) | 13 | 4 | — | AZ.91 | DTD 281A | 5·0 | 14·0 | 7 | 55 | 2·5 |
| | | | — Do. — | | | | | Do | 8 hrs. 210° C. | (6·5) | 13 | — | — | AZ.91 | DTD 285A | 7·0 | 14·0 | — | 80 | 0·9 |
| 2·5 | — | R | — | — | 0·1 | — | — | None | None | (3) | (10) | (5) | — | AZ.31 | (AZ.31) | 3·0 | 11·0 | 8 | 45 | 4·8 |
| 3·5 | 0·1 | | 0·3 | 0·05 | 0·4 | 0·01 | 1·5 | | | | | | | | | | | | | |
| 2·5 | — | R | — | — | 0·1 | — | — | None | None | (4·0) | 9 | 4 | — | Magnuminium | DTD 350A | 4·0 | 10·5 | 5 | 45 | 1·6 |
| 4·5 | 0·1 | | 0·3 | 0·05 | 0·4 | 0·01 | 0·10 | or annealed | | | | | | 299 | | | | | | |
| | | | Sn 4·5—7·5, Ag 0—2·0 | | | | | | | | | | | | | | | | | |
| — | — | R | — | — | 1·0 | — | — | None | None | — | 6 | 3 | — | AM.503 | DTD 140B | 1·5 | 6·5 | 4 | 40 | 5·0 |
| 0·08 | 0·02 | | 0·05 | 0·03 | 2·0 | 0·005 | 0·03 | | | | | | | | | | | | | |
| **GRAVITY DIE CASTINGS** | | | | | | | | | | | | | | | | | | | | |
| 7·5 | — | R | — | — | 0·15 | — | — | None | None | (4·5) | (12) | (4) | — | A.8 | DTD 59B | 4·5 | 12·0 | 4·5 | 60 | 1·1 |
| 9·0 | 0·15 | | 0·3 | 0·05 | 0·4 | 0·01 | 1·0 | 4–8 hr. 250°C. | | (4·5) | (12) | (4) | — | A.8 | DTD 59B | 4·7 | 12·5 | 4 | 65 | 0·9 |
| | Sn 0·10 | | Cu+Si+Fe+Sn+Ni—0·40 | | | | | | | | | | | | | | | | | |
| | | | — Do. — | | | | | 12 hrs 435° C. | None | (4·5) | (15) | (10) | — | A.8 | DTD 289A | 4·5 | 18·0 | 12 | 60 | 4·0 |
| | | | — Do. — | | | | | Do. | 8 hrs. 210° C. | (6·5) | (15) | (3) | — | A.8 | A.8 (S.P.) | 6·5 | 16·0 | 4 | 75 | 1·3 |
| 9 | — | R | — | — | 0·15 | — | — | No ne | None | (5.0) | (11) | (2) | — | AZ.91 | DTD 136B | 5·5 | 11·5 | 2·5 | 60 | 0·5 |
| 10·5 | 0·15 | | 0·3 | 0·5 | 0·4 | 0·01 | 1·0 | 4 hrs. 250°C | | (5·0) | (11) | (2) | — | AZ.91 | DTD 136B | 5·7 | 12·5 | 2 | 70 | 0·5 |
| | Sn 0·10 | | Cu+Si+Sn+Fe+Ni—0·40 | | | | | 16 hrs. 435°C. | | | | | | | | | | | | |
| | | | — Do. — | | | | | | None | (5·0) | (14) | (5) | — | AZ.91 | DTD 281A | 5·5 | 15·0 | 18 | 60 | 2·7 |
| | | | — Do. — | | | | | Do. | 8 hrs. 210° C. | (7·0) | (14) | (2) | — | AZ.91 | DTD 285A | 8·0 | 15·0 | 2 | 85 | 0·7 |
| 2·5 | — | R | — | — | 0·1 | — | — | None | None | (4) | (12) | (6) | — | AZ.31 | (AZ.31) | 4·0 | 13·0 | 10 | 50 | 5·2 |
| 3·5 | 0·1 | | 0·3 | 0·05 | 0·4 | 0·01 | 1·5 | | | | | | | | | | | | | |
| 2·5 | — | R | — | — | 0·1 | — | — | None | None | (4) | (10) | (5) | — | Magnuminium | DTD 350A | 4·0 | 11·0 | 7 | 50 | — |
| 4·5 | 0·1 | | 0·3 | 0·05 | 0·4 | 0·01 | 0·10 | or annealed | | | | | | 299 | | | | | | |
| | | | Sn 4·5—7·5 Ag 0—2·0 | | | | | | | | | | | | | | | | | |

# Automatic Control of Soaking Pits

By C. H. Stone

*The smooth and organised working of soaking pits is a primary factor in steel mill production and yield, and as such deserves considerable study and attention. With this in view the author briefly reviews requirements of socking pits, their design and type according to the fuel to be used, and particular attention is directed to the heating cycle with the object of instituting controlled conditions in the pits to facilitate work in the mill.*

## The Purpose of the Soaking Pit

THE steel plant or, more specifically, the melting shop produces steel in furnaces from which it is tapped into moulds and allowed to solidify. These casts, or ingots, represent therefore, the product of the melting shop. The weight, form, and quality depend upon metallurgical and economic considerations—mill and plant layout, and type of product required. The ingots from the furnace, forming the cast, reach the soaking pits in a semi-molten or even cold state. First, they must be stripped from the moulds, when they are ready for the next stage of production—treatment in the soaking pits—after which they proceed to the mill and to the first stage of cogging.

The mill, being a continuous process, essentially requires a constant supply of ingots in proper condition for rolling. It is, therefore, the first duty of the soaking pits to provide ingots in right rolling condition. From this, another point arises—storage capacity. The soaking pit acts as a buffer between the intermittent production from the melting shop and the continuous demand of the mills, and must, therefore, be elastic in its capacity to accept the products of the melting shop as well as to supply properly treated ingots to the mill.

If one considers the cooling of ingots, or the freezing of the metal in the mould, from the walls to the core, one can readily understand the difficulty in maintaining the balance between supply and demand at the soaking pit. There is the added complication that certain steels require cooling right down to the solid state and preheating in the pits as dictated by their structure. The aim of the heater is to bring the ingot from its stripped stage, semi-molten inside, solid and cold at the edges, and solid but red-hot on its sides, to a uniform temperature throughout by thorough soaking in the pit, i.e., soaking in its true sense.

The soaking pits must, therefore, fulfil the following requirements :

(*a*) Proper design to meet all the heating cycles required by the varying qualities of the steels.
(*b*) Adequate capacity to balance supply and demand.
(*c*) Disposition in relation to auxiliary plant so as to assist rather than to impede the smooth flow of ingots through the pits.

## The Pit Itself

The design and type of pit demands primarily upon the fuel used. Fuel, therefore, should be considered first of all :

(*a*) Since the temperature range is between 1,200° and about 1,350° C. high flame temperatures are not required.
(*b*) As the process is a reheating and soaking cycle, fierce flames are dangerous.
(*c*) This danger is aggravated by the fact that the flames come unavoidably into contact with the surfaces of the ingot.

Whichever fuel is employed these three facts must be taken into account and, therefore, the design of the pit and combustion conditions must be considered with reference to them.

(A) Of all the economic fuels in this country, blast furnace gas is the most suitable from the above points of view.
(*a*) The flame temperature is just high enough.
(*b*) The flame is soft and long.
(*c*) Combustion is easily controlled and can be made uniform so that there is, therefore, little danger of over-heating at the surface of the ingot.
(*d*) The quality of the gas does not vary greatly. The only item which requires watching is its moisture content ; in most plants, however, the quality of the gas reaching the pits is reasonably uniform.

Should quicker heating be permissible, on some casts, a small addition of coke-oven gas, during reheating only, will increase the calorific value without upsetting the volume, so that flue design can be kept in conformity with optimum velocity conditions.

Taking, for example, a B. F. gas-fired pit designed for a 70–90 ton charge, 189,000 cubic feet or 18,000,000 B.T.U's. per hour are used for heating up. By the addition of 10,000 cubic feet per hour only, there is a gain of 5,000,000 B.T.U's. per hour or an increase of 27.7%.

In most cases, however, this will not be necessary, since the heating cycle on blast furnace gas alone is metallurgically as well as economically sufficient.

Another factor is that of heat efficiency. Blast furnace gas scores over all other fuels in that heat recovery can be used to the fullest extent, since the air for combustion, as well as the gas, can be preheated without reaching flame conditions detrimental to the process.

The fuel, being clean and cold, can be controlled with accuracy, so that good combustion can be achieved over the range required. For this reason also, control of the atmosphere (in this case, reducing) can be undertaken without compromise.

The pits used with blast furnace gas are of the orthodox regenerative type, with gas and air preheating checkers, or in the modern Isley Morgan type with air preheating only in a multiple checker system.

(B) The next fuel to be considered is the mixture of blast furnace and coke-oven gas. The calorific value is usually of the order of 230-250 B.T.U's. per cubic foot, but may sometimes reach 300 B.T.U's per cubic foot.

Owing to this higher calorific value we now have to

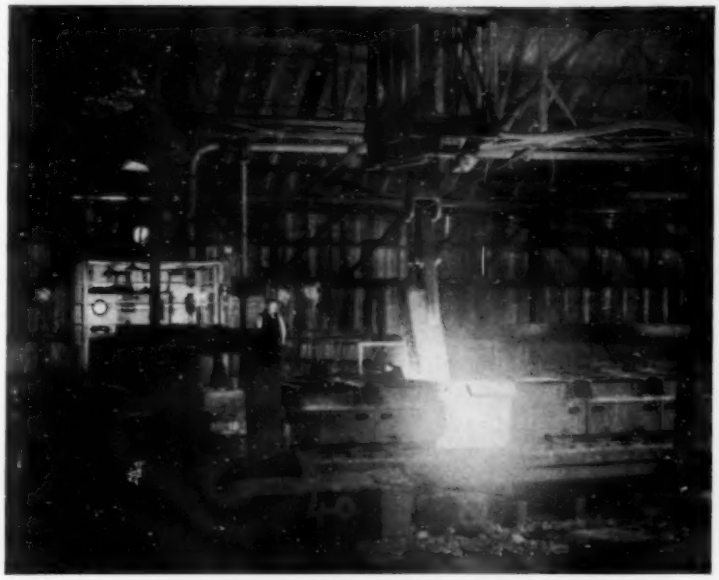

**Fig. 1.—Typical soaking pit with control room in the background.**

consider those types of pits in which preheating of the air only is required and, therefore, the recuperative pit is better suited and more economical.

More careful flame direction is essential and, from this point of view, various designs have been developed, of which the Surface Combustion and Amsler Morton are the more modern types known in this country.

As the flame temperature can become critical, flame direction is very important and, consequently, more rigid maintenance of ports or burners is essential. It is also essential to secure accurate control of the proper gas mixture. The volume of coke-oven gas may be reduced when the soaking period is reached without great interference with the flow conditions owing to the relatively small change in total volume for the reduction in calorific value. An economic heat recovery is still possible. Atmosphere control can be achieved without difficulty since the cleanliness of the gas and its uniform temperature render accurate measurement possible.

(C) Coke-oven gas used alone gives a short fierce flame (particularly when, as in this case, there is no preheating) and, therefore, the introduction of pits with multiple burners is essential, as dictated by considerations of flame direction and the distance of the ingots from the flames. Air preheating is generally omitted owing to the nature of the gas and its high calorific value ; it may, moreover, introduce complications with little return for the necessary initial outlay and space occupied.

We have, therefore, a furnace without gas or air preheating and with multiple burners. The firing may be from the bottom upward surrounding the ingots, tangential introduction of the flames as with the Salem pit, or an arrangement of multiple burners fired from both sides. There is no heat recovery but, in compensation, good combustion is secured since accurately designed burners are used.

(D) There now remains producer gas, the most commonly employed fuel in Britain for soaking pits. This is, of course, crude producer gas, i.e., gas uncleaned and containing tar and other constituents, at a temperature of 400° to 600° C. at the gas control valve.

In comparison with present-day practice, the use of this gas, in its crude condition at least, is no longer as economical as it used to be. With a calorific value of 140–160 B.T.U's per cubic foot, we have again to consider gas and air preheating and come back, therefore, to the orthodox regenerative pit. As gas and air preheating can result in very high flame temperatures, strict attention should be paid to design and control. The flame is soft when reducing, but very fierce when fired with excess air, especially at high preheats. The preheating is generally obtained by means of regenerators and care must be taken not to overheat the gas. Restricted checkers are, therefore, unavoidable and stack losses high. The measurement of crude producer gas flow involves considerable difficulty and can only be undertaken with special precautions, so that good control is doubtful, and is best achieved on the basis of temperature measurement, the valve position being adjusted by the pit temperature rather than on a volumetric basis. Therefore, atmosphere control becomes difficult, aggravated by variations in the calorific value of the gas.

For these very reasons, automatic control is absolutely essential if results are to be obtained comparable with other fuels. Even so, more and closer attention is necessary with this type of fuel. To obtain with crude producer gas-fired pits costs, reckoned in shillings per ton, comparable with those obtained with other fuels requires good heaters, good controls and, above all, efficient management.

It will probably be more economical for individual plants, especially from a national point of view, when the use of cleaned producer gas becomes extended, the gas being produced from coke or coals of lower calorific value, while the better class coals with high volatile contents are used in plants where their special qualities can be exploited and the by-products can give a return. A clean gas of, say, 120–130 B.T.U's per cubic foot, would relieve the anxieties of the heater and of the management, and the products, i.e., the ingots, could be better treated, and the losses and repair costs would be reduced, combined with far better control. Everybody would gain—nobody would lose. Even the producer men would be glad to forget the dirty task of burning out mains and, simultaneously, wasting the most valuable constituents of the fuel.

In these notes on fuels, it is not intended to deal with oil as a fuel for soaking pits in this country as, under present economic conditions, its use is unjustified. From an engineering point of view there would be no gain by cost reduction to warrant the use of this expensive fuel, while its availability as such is not unlimited.

Generally, it can be truly stated that with present-day knowledge of fuels it is a fallacy to demand a good fuel but to have little or no means of control over it ; it is better to have a fuel, poorer but stable in quality, easy to measure and, therefore, susceptible of control. This is preferable, obviously, to the common practice of using

a good quality coal but having no means of exploiting these very qualities. In this age of mechanisation and scientific advancement it is surely incongruous to fill a gas machine with a soft through coal—varying from dust to 4 in. lumps or more.

To all deeply concerned with efficiency it is clear in designing new plants or replanning old that the type of fuel and gas-plants should be chosen, not on the basis of high B.T.U. content in the exit gas, but from the viewpoint of the ultimate employment of the gas at the production unit, i.e., the furnace. If the latter is of modern design as regards burners, flues, etc., one can be assured of a high degree of efficiency with a stable low calorific value gas (cold and clean); why, then employ a high-class coal to produce a gas which, because of its temperature, variation in quality, dust, soot, and tar disposition, prohibits the use of accurately designed burners and valves, involves much compromise in combustion efficiency in meeting fluctuations in demand and eventually results in high stack losses, lower efficiency, higher maintenance of brickwork, and air pollution.

Possibly the increase in the cost of fuel and the limitation of available resources will enforce a realisation of these facts.

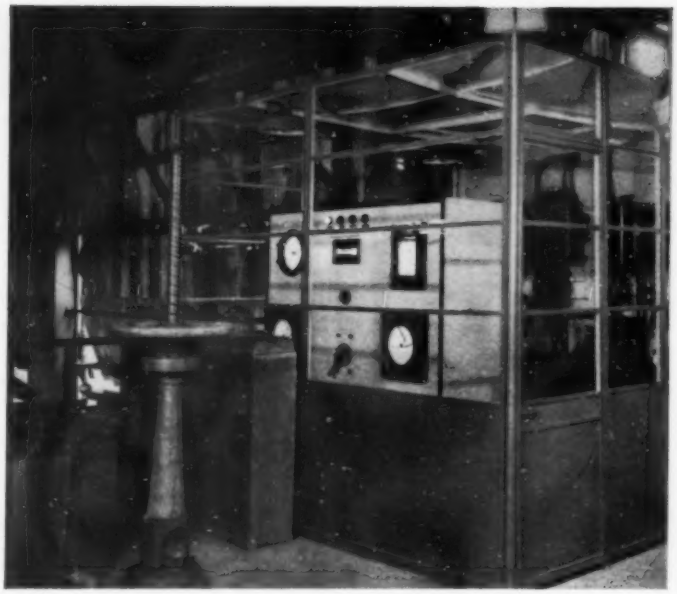

**Fig. 2.—Control room for soaking pit showing regulators mounted at rear of control panel.**

### General Points Concerning Pits

The layout of the pits should be such that they are readily accessible both to production and maintenance personnel, and that repairs or rebuilds do not interfere with the charging and discharging of pits, nor with the transport of ingots. The building itself should provide protection from air currents on the checkers so that wind direction does not interfere with preheating. If the layout is properly arranged, fewer hands will be required to do an efficient job and have more time to do things correctly.

Soaking pits are, of course, production units, but should not be judged in terms of tons per hour. The essential point is to have more units than are required on average so that they can fulfil their function as *soaking pits* and preserve the necessary elasticity so vital in their duty as a buffer. The heater will then never have to compromise between the requirements for efficient soaking and the demands of the tonnage men at the mill, who hate waiting for a heat. The heater is, thus, often placed in a difficult position. How desirable to have an empty pit on slow gas available for an extra fast cast from the melting shop! How galling to strip hot ingots and stack them carefully upright—not in a heap—no room being available in the pits!!

Lids usually constitute the main item for maintenance, and are responsible for more than is apparent at first sight. Tight-fitting lids permit the running of the pits on a slight pressure, with the result that there is better control over the rate of heating, combustion efficiency, and the checkers. Once leakage commences, the suction must be increased or the gas reduced. Increase of suction draws in air with its inevitable evils, while reduction of gas affects combustion as well as the atmospheric conditions and, of course, air infiltration occurs to compensate for the reduced volume input.

The condition of the lids is, therefore, of paramount importance, and their proper maintenance is amply repaid. It is not intended here to discuss the merits of dry or liquid slagging; slag, up to a point, is unavoidable, but, by the time the pits are run with only 0·3% or less iron loss in the slag, dry slagging will be found most convenient. The pit can then be kept in production for reasonable periods and the time taken for slagging employed for port cleaning and patch repairs wherever necessary.

### The Heating Cycle

The ingots varying in temperature condition are placed in the pit which may itself be right up to temperature. The gas and air are then fully turned on and the temperature brought up to its maximum required value and maintained until ready to draw. All this is, generally, judged by eye and, of course, depends entirely upon the skill of the heater, aided solely by judgment and experience. This method has been effective in the past, but is it the best and only way?

Now, we know that the temperature should be increased at a certain rate, say, for argument's sake, 100° C. per hour. The heater sets his gas valve roughly to cope with this. How roughly depends upon his judgment. When he thinks he is up to temperature, he first of all reduces his air and then starts setting his gas down. With the means at his disposal this is his safest way. He may even reduce his stack draught—but this is not so common as one might think. Usually, his valve gear will not stand up to engineering examination, let alone accurate handling.

In order to determine what the heater really ought to do if he had his judgment backed up by facts, i.e., measurement, and so was guided in his actions, let us examine a blast furnace gas-fired pit of the orthodox regenerative type, but fully automatically controlled.

The heating rate is given by applying, say, 180,000 cubic feet of gas per hour for the particular type of steel.

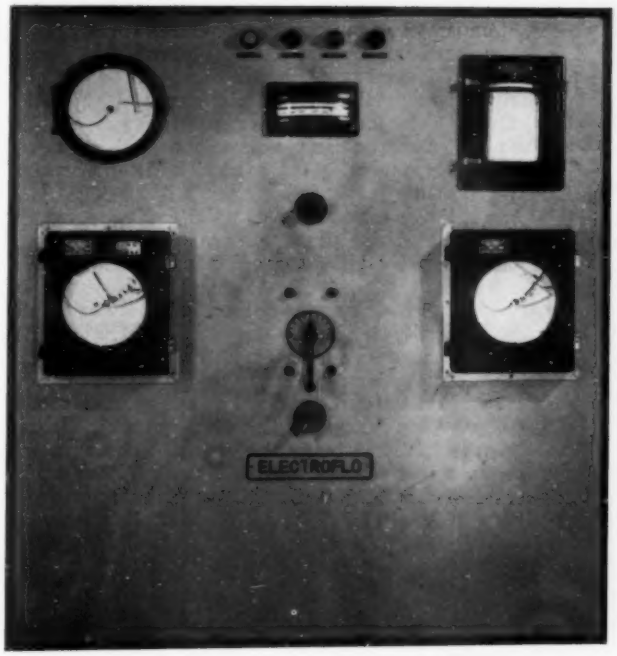


**Fig. 3.—Control panel showing temperature, pressure, gas and air-flow recorders, and temperature regulator with hand control and pilot lamps.**

The heater sets his gas valve by hand until the rate-of-flow recorder shows him 180,000 cubic feet per hour. He now leaves the pit. According to careful tests on this pit, it has been found that, say, 0·75 cubic feet of air per cubic foot of gas gives the correct skin (or scale) on the ingot, to fulfil the mill requirements. This skin drops off in flakes after the first two or three passes at the cogging mill, and the clean ingot is broken down.

The automatic regulator measuring the gas and air is set to provide this ratio and maintains it. The temperature regulator opens its gas valve fully but, due to the setting of the hand control valve, passes 180,000 cubic feet of gas per hour, and no more. We have, therefore, an input of 180,000 cubic feet of B.F. gas plus 135,000 cubic feet of air per hour. This volume, after preheating, enters the pit. Now, since we do not want any air to be drawn in, the stack damper should be set in such a position as to withdraw this volume of combustibles only. By previous tests one may have found that *at the point of measurement* in the pit a suction of, say, 0·045 ins. w.g. ensures this condition. Therefore, the pit pressure regulator is set to control at this figure and will cause the damper to float so as to maintain it. The stack pull is now under control and is only allowed to discharge the volume that will maintain the required " sting-out " at the lids. The judgment of the " sting-out " is not easy, as it depends upon the quality of the gas, type of combustion, etc., but it is easier to judge on soaking pits than on, say, open-hearth furnaces.

This pit is now, truly, under control. Any change in conditions is measured and the respective regulator takes the appropriate corrective action. The pressure regulator is aware of any change in input and immediately responds. As the temperature increases in the pit, the required conditions are successively maintained. The heater simply glances at the temperature record and knows how he stands. The rest is seen on the gas and air flow charts and, of course, on the pit pressure recorder. When the required temperature is approached, the temperature regulator comes into action and gradually reduces the gas supply, while the ratio regulator simultaneously reduces the air supply in proportion, these changes being automatically recorded on the flow charts. The damper is correspondingly adjusted by the pit pressure regulator so that the volume of gases discharged to the stack is also reduced. The temperature now follows a straight line at the set figure and, after two or three hours soaking, the heater looks at the chart, opens the lids to assure himself that the regulators have done their job, and has his pit ready for drawing. During this period reversals have been carried out on a basis of a predetermined time cycle; this is usually sufficient since there are no reactions to interfere with combustion, no one-sided pull possible, and no clogging-up of checkers, conditions being practically even. A check-up with thermocouples in the out-going flues can be arranged and the records examined for any suspicion as to conditions of valves, ports, or any other item.

During the above heating cycle, the flow of blast furnace gas will have changed from 180,000 cubic feet per hour to, say, 60,000 cubic feet per hour, the flow of air from 135,000 to 45,000 cubic feet per hour, and the damper shifted from, say, 14 in. to about 4 in. from the bottom of the flue. This has all been done entirely automatically by measuring and controlling the various quantities through their respective valves or dampers. Cast to cast, day in and day out, all these quantities are accurately measured and correctly adjusted. It is no wonder that the heater comes to regard the controls as his friends and that the mill manager becomes proud of the yield he achieves.

The roller man in his cabin looks forward to rolling these ingots as his judgment is simplified. He knows these ingots are soft, with no twisting nor squeezing through the mill, and causing no sudden demands on the mill engine. There is smooth plant operation with consequently improved organisation.

Now, the regulators described above have really done what the heater himself should have done. Could this reasonably have been expected of him? Anyone, knowing the heater's job, realises that he could not possibly have done so. Sometimes, he is able to adjust eight times during soaking, sometimes four times, depending upon his jobs in hand. Even when he alters his settings, he is going by trial and error. Tests carried out on soaking pits by measurements only—leaving the heater to use his own judgment—show this up well.

(*a*) He is bound to overheat at the beginning of the soaking period unless he is right on the spot. Even then, he may take off too much gas and, at the best, can only approximate the desired temperature by following a zig-zag record.

(*b*) Adjustment of the gas screw is not sufficient. He must attend to the air screw as well—leaving another chance of error.

(*c*) There is also the pressure in the pit to be considered. By cutting down his input, ought he to follow with the damper? Obviously, but again, by how much?

(*d*) He is, therefore, reduced to a compromise, and takes air off, usually by stopping the fan, lowers the damper a little, and proceeds to the next pit.

Invariably, the damper is not low enough and the stack draught is more than enough to exhaust the products of combustion; consequently make-up air is drawn in through the lids. This air strikes down on the top of the ingots and meets the highly reducing flame. Combustion now starts in the upper region of the pit, at the outgoing port and even further. And so the trouble starts. Early repairs, damaged ports, slag in the ports, erosion of brickwork and high iron loss in the slag.

However, up to now, things have apparently gone quite well, the heaters have done their best, and tonnage has been produced, so why the need for automatic controls. But is this actually the case? Have the heaters been able to do their best work, and are costs as low as they might be? If it is possible to get down to 0·3% loss in the pit and maintain a cleaner pit for a long period, if the repair cycle of the pits can be reduced and if the heater can be assisted so as to have more time for attention to detail, surely means to achieve these ends must be justifiable. Capital expenditure will certainly be incurred, but the above returns will be the result.

It may be argued that the many alterations in design which have been made from time to time have not yielded the results expected. This is, mainly, due to the fact that conditions were not completely known. Without measurements, the results cannot properly be analysed. There is too much interplay of variables—which, unless harnessed before the alterations are undertaken, will invalidate judgment.

### Essential Features of the Controls.

From the foregoing, it will be clear that there are three quantities which must be controlled automatically.

(*a*) *Pit Pressure:* This is the most important, though the least understood and, in consequence, the least conspicuous item of control. This requires not only a good sensitive regulator (sensitive on account of the infinitesimal static values which have to be controlled) but also a good well balanced control damper and, of course, a thoroughly investigated and understood measuring point in the pit. At this point, readers should be warned not to accept the figures given as being applicable to every plant. What suits one job is unsuitable for another.

(*b*) *Temperature Control:* This requires to be a sturdily constructed steelworks regulator and not a laboratory instrument. Upon it depends a soaking pit containing 80 or more tons of steel. It must be of the "fully-floating" type, since huge volumes of gas and air cannot be shut on and off or their rates of flow suddenly changed. It must be accurate and simple to maintain, and flexible in its adaptation with no fixed characteristics, such as cams, etc. It should be rapid in operation and with ample reserves of power to move heavy valves liable to stick or jam, derived from an independent or positive source. The location of the point of measurement must be carefully chosen, and be representative of the actual condition to be controlled.

(*c*) *Gas-Air Ratio Control:* This is the simplest of the three controls and is the best known. Here again, reliability and sturdiness are essential.

For the purpose of supervision, recorders for temperature, gas and air volumes, and furnace pressure are necessary.

This valuable equipment for the soaking pit should be properly housed, maintained, and kept in clean condition. Owing to its importance for the efficient operation of the pit, it should occupy a prominent and accessible position, and not be relegated to some odd corner where it may gradually be overlooked.

Automatic control can achieve the optimum results from the oldest design of pit, whereas new design alone is no guarantee of efficient performance. This is the reason why more than 90% of recently laid-out pits in America are operating under full automatic control and, thereby achieving the fullest benefit of the new designs. One of the reasons why, in this country, we have no clear cut results attributable to modern design is that the new pits are run on the same method of compromise as the older plant. This brings about, in consequence, the impression that nothing much is to be gained by modern pit design, and the anomaly that while in some works the new design is beneficial, in others nothing much can be shown in favour of the new design. Briefly, there is no basis for comparison since the performance is not under control but is dependent upon the personal judgment of those in charge.

There exist a few reliable and sturdy makes of control equipment on the market, suitable for our steel plants, and which have been proved equal to the job. The instrument manufacturers have provided the gear and have studied the problem. They know enough to be of great assistance and will play their part in the coming reorganisation of this basic industry.

### Conclusion

The above is an attempt at a brief summary of the subject under review. It has been written in this way, purposely, so as to leave details open—details associated with the plants and sites which provide the proper platform for their discussion. The soaking pit is one of the most, if not the most, important unit in the steel industry and incurs a tremendous responsibility as to its products. It can, and often does, spoil the efforts and labours of the melting shop. Its smooth and organised working is the primary factor in mill production and yield, and as such deserves the greatest study and attention.

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## Electrochemists Congress in Canada

ELECTROCHEMISTS from all parts of the world met in Toronto on October 16–19, 1946. The general theme of the Congress, organised by The Electrochemical Society, was "Electrochemistry's Contributions to the Winning of the War," and sessions were held on: Advance in Electrodeposition, Rectifiers and Power Supply, New Plastic Insulators, Theoretical Electrochemistry, the New Batteries, and Problem in Magnesium Production. Some forty scientific and technical reports were presented by experts in the various fields.

An important event at this Congress was the presentation of the Edward Goodrich Acheson Gold Medal and Thousand Dollar Prize to Professor H. Jermain Creighton of Swarthmore College, Pennsylvania, in recognition of his outstanding accomplishments and fundamental researches in electrochemistry. In his speech of acceptance, Dr. Creighton revealed a number of far-reaching inventions and discoveries in electrochemistry made during the war.

# The Iron and Steel Institute

## Annual Autumn Meeting Held in London

A VERY comprehensive programme was arranged for the Autumn Meeting of The Iron and Steel Institute, held at The Institution of Civil Engineers on November 13–14, 1946. Apart from about half an hour devoted to official business at the opening session, morning and afternoon sessions of both days were devoted to the presentation and discussion of reports and papers. In addition there was a film display at which one film recorded flame characteristics and other aspects of trials and experiments conducted in the open-hearth melting shop at Templeborough; a second concerned the control and prevention of distortion in arc welding; a third illustrated the brightness of liquid steel streams; and a fourth gave a record of the technique of the quick-immersion thermocouple, showing the method of assembly and the ways in which it has been applied in different works and types of furnace.

### Technical Sessions

Morning and afternoon technical sessions were held in the Lecture Theatre of The Institution of Civil Engineers on each of the meeting days. At the first two sessions various aspects of the report on "The Influence of Port Design on Open Hearth Furnace Flames" were discussed. This report, known as Special Report No. 37, and comprising 187 pages of text, 17 plates and numerous tables and drawings, describes a collaborative research, organised by the United Steel Companies, Ltd., and the British Coal Utilisation Research Association, carried out by a team of workers, to obtain information on the combustion processes, heat transfer and gas flow by studying the effect of different port design.

At the third session three papers were presented for discussion; one dealing with the origin and constitution of certain non-metallic inclusions in steel, another with the neutralisation of sulphur in cast iron by various alloying elements, and a third concerning the removal of hydrogen from steel. At the fourth session a descriptive paper was presented giving detailed information of the new annealing plant for steel strip in coils at the Whitehead Iron and Steel Co., Ltd., Newport, Mon. Then a further three papers were presented for joint discussion on the subject of protection of iron and steel against marine corrosion and fouling.

The papers presented together with the various discussions are too voluminous to present here, in any case, advance copies of the papers are available, and a complete report of the proceedings will be published by The Iron and Steel Institute. It is thought, however, that a brief summary of the various work discussed will be helpful in assessing the importance of this meeting.

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### THE INFLUENCE OF PORT DESIGN ON OPEN HEARTH FURNACE FLAMES.

By J. H. Chesters, D.Sc. and M. W. Thring, M.A., F.Inst.P.

THE lack of fundamental information on the combustion processes in the laboratory of the open hearth furnace makes it impossible to design a furnace in the sense that a bridge is designed. The purpose of the trials described in the Special Report No. 37, which were carried out by a team of workers in a collaborative research organised by the United Steel Companies, Ltd., and the British Coal Utilisation Research Association, was to obtain some of this information by studying the effect of different port designs on the combustion, heat transfer and gas flow. This in turn was to be correlated with output ratio.

Three furnaces of very different type were chosen in one melting shop and several trial casts controlled as closely as possible were made on each furnace to allow comparisons to be made. External factors, such as the gas and air flow, gas quality, preheat temperatures, gas pressures, $CO_2$ in waste gases, and metallurgical load, were carefully measured, and heat flow meters (described in a separate paper), a narrow-angles radiometer, roof pyrometers, etc., were used to study the internal radiations. Preheat and waste-gas temperatures were recorded, using suction pyrometers, and the factors affecting preheat were analysed. The flame location, velocity and stability, together with the rate of mixing of the gases, were studied by means of slow-motion cinematograph films and by a multiple sampling probe inserted through the roof.

The purpose and plan of the experiments were presented in an introductory address by Dr. Chesters, based on Section I, Parts 1 and 2, and Section II, Part 8, of this Special Report. These aspects were later given special consideration and subsequently discussed. Thus special instruments and technique, based on Section I, Part 3, and Section II, Parts 1 to 7, 11, 13 and 14, were introduced by Dr. Chesters; thermal conditions within the open hearth furnace, based on Section II, Parts 10 to 14, and Section III, Part 2, was introduced by Mr. Thring; and general conclusions, based on Section II, Part 9, and Section III, Parts 1 and 2, were given by Mr. W. Geary.

### A HEAT-FLOW METER FOR USE IN FURNACES.

By R. H. Baulk, M.Sc., and M. W. Thring, M.A., F.Inst.P.

THE modern trend in assessing the performance of a furnace is to regard thermal efficiency as an important criterion. The thermal efficiency of a furnace depends almost entirely upon two factors: (*a*) The fraction of of the heat released by combustion which is lost by radiation through openings and by conduction through brickwork, and (*b*) the fraction of heat released by combustion which passes out of the heat-utilising region as sensible heat in the products of combustion.

Heat is transferred from the flame to the charge by radiation and convection. The heat-transfer coefficient thus corresponds to the sum of two effects. Improvements in the radiation heat transfer with a given temperature-difference between the flame and the

charge can only be obtained by increasing the emissivity of the flame, since the furnace designer cannot control the absorptivity of the charge. Improvements in convection can be effected by increasing the velocity at which the gases impinge on the charge.

Convection is therefore largely limited by the erosion resistance of those refractories which serve to maintain the requisite velocities. Hence the greatest improvement in furnace firing will probably arise from the discovery of new methods of increasing flame radiation.

The development of an instrument for measuring flame radiation is the first step towards the investigation of methods for increasing it. Such an instrument would enable an assessment to be made of the effects on the radiating power of the flame of : (*a*) The water vapour and carbon dioxide content, (*b*) delayed combustion (which gives rise to soot formation), and (*c*) the addition of hydrocarbons. It is virtually impossible to predict the effect of these factors with any accuracy by calculation, both because of the large number of variables and also because many simplifying assumptions have to be made before mathematical calculation is possible. On the other hand, it appears probable that interesting effects would be observed if a furnace were operated so that only one variable at a time was changed, provided that an experimental technique were available for direct measurement of the heat-transfer coefficients.

It is shown to be useful to measure the heat flowing upwards by radiation from the charge and that flowing downwards to the charge separately and to obtain the difference by subtraction. A further advantage of an instrument which can read the upward and downward radiations separately is that it can be used also between the crown and the flame so as to give the total heat radiated back into the furnace by the crown as well as the net heat flowing into the crown.

The heat transfer over the hearth area of a furnace should vary as little as possible, and an instrument exploring this variation would be invaluable in designing a furnace so as to make the best use of the available heat. In particular, if it could be used in conjuncton with a furnace in which all the steelmaking variables were kept constant, the effect of variations of heating power upon output could readily be found.

Since radiation is of greater importance than convection in high-temperature furnaces, it was decided to concentrate first on an instrument which would indicate the radiation upwards and downwards without taking account of effects due to convection. Although these effects may be an appreciable fraction of the transfer they are unlikely to upset any broad conclusions drawn from the work.

The work on such an instrument has reached a stage where a practical research tool has been evolved and used to compare the heating power of different flames, to examine the effect of flame thickness upon the heat flow from the flame, and to compare the heat flow at different points along and across the flame. It has shown, for example, that measurement of the downward heat flow below the flame at its two ends gives a valuable quantitative measure of the effective flame length ; in the case of very slow combustion the downward heat flow at the outgoing end is greater than at the ingoing end. It has also been shown that the heat-absorbing area of the surface of the charge is not very fully utilised either in the steel furnace or in the glass tank, the former because the flame does not cover the whole area and the latter because the cold batch is not spread out.

## THE ORIGIN AND CONSTITUTION OF CERTAIN NON-METALLIC INCLUSIONS IN STEEL

By J. R. Rait, B.Sc., Ph.D. and H. W. Pinder, Assoc.Met., F.I.M.

IN most steels the presence of non-metallic inclusions is undesirable, because they are often a potential source of weakness, whether the steel in which they occur is being stressed statically or dynamically. In addition, inclusions in steel are the cause of many rejections on mechanical testing, especially when the test pieces are prepared transversely to the grain flow of the material. The main sources of these non-metallic inclusions are :—

(1) Deoxidation, and the segregation of the products of deoxidation.

(2) The presence of sulphur and phosphorus, and the segregation of their compounds.

(3) Extraneous sources.

(1) Deoxidation is no doubt responsible for some unavoidable inclusions in steel. However, steel-melting and deoxidation techniques are now sufficiently well developed to enable large quantities of such inclusions to be avoided. Difficulties may, however, be encountered in large ingots and also in badly designed ingots, where segregation renders the deoxidation products much more harmful, while low casting temperatures will prevent the escape of many inclusions into the part of the ingot normally discarded. In the present paper attention will be focussed on the effect of aluminium, which is widely used as a final deoxidant and for controlling the grain-size, as its injudicious use can lead to a type of inclusion which is very troublesome in machineing and drilling.

(2) The sulphur content of the finished metal can be controlled in all steel-making processes. This is accomplished by special selection of the charge materials and the fuel, as in the acid process, or the sulphur (and phosphorus) can be partially removed by chemical methods, as in the basic process. Serious sulphur segregation or inclusions were encountered only on rare occasions during this investigation, and inclusions of this type will therefore be ignored.

(3) Although the formation of some non-metallic inclusions is inevitable in ridding the molten steel of its gaseous and soluble constituents by converting them into solid particles, there are many other sources which merit even more careful study. During production, molten steel and slag must come in contact with a large number of refractory materials, such as the furnace lining, the launder lining, the ladle lining, stoppers, and nozzles, tundish linings, and, in bottom-pouring, trumpets and runners. As a result of their erosion and corrosion by the molten slag and metal, these refractories offer a very real and ever-present source of non-metallic inclusions ; evidence of such fluxing can be readily noticed on visual examination of the mentioned refractories after a heat of steel has passed over them. In addition to physical erosion, the slag chemically attacks the launder and ladle linings and, in the case of tilting furnaces (where slag is allowed to pass into the ladle from the commencement of the top), some of the slag and its reaction products may be trapped in the steel.

By far the larger number of inclusions and segregates found are of extraneous origin, and it is therefore with these inclusions that this paper is chiefly concerned. The term " inclusions of extraneous origin " covers all

inclusions introduced into the steel as a result of:

(*a*) The trapping of the slag.

(*b*) The trapping of corroded and eroded refractories used at any stage in the steel making.

(*c*) The trapping of the products of reactions between the steel and the refractories with which it comes in contact.

The authors' review the phase diagrams relating to the study of non-metallic inclusions in steel, and examines the important ternary system $MnO–Al_2O_3–SiO_2$; the most probable phases occurring in the complex system $CaO–MgO–FeO–MnO–SiO_2Al_2O_3$ are described in the light of the review. A large number of different types of inclusions in acid open-hearth and basic electric-furnace steels (direct-poured, tundish-poured, and bottom-poured) are identified by extraction and by the use of phase-diagram data, chemical analysis, metallographic examination, and X-ray examination.

It has been possible to ascertain the sources of many of these inclusions with a fair degree of accuracy, and a table summarising these results is given. Practical suggestions are made for reducing the incidence of harmful non-metallic inclusions in steel.

## THE NEUTRALISATION OF SULPHUR IN CAST IRON BY VARIOUS ALLOYING ELEMENTS.

By H. Morrogh.

PREVIOUS work by the author has shown that sulphur is an important alloying element in cast iron and, in addition to its direct carbide-stabilising effects, has other major influences. Undoubtedly, however, it has undesirable features and this has led to its almost universal acceptance as an unavoidable evil and hence its amount is usually kept to a minimum. This principle has served and will continue to serve a useful function in general iron-foundry practice, but for the fuller development of the potentialities of cast iron it will be necessary to regard sulphur as a useful alloying element.

In many respects sulphur as an alloying element is unique in so far as it can occur in many different forms as sulphides of other alloying elements, e.g., manganese, titanium, zirconium, vanadium, copper, chromium, aluminium, etc. The effect of sulphur in cast iron depends almost entirely upon the manner in which it is combined with another element, that is, upon which sulphide is formed. The elements nickel, molybdenum, copper, chromium, and aluminium, all form fairly stable sulphides, and it was the object of the work here reported to determine to what extent each of these elements could combine with sulphur in cast iron. A base iron with a relatively low-manganese content (pig iron *A*) was chosen for this investigation; in addition, some experiments were carried out with a Swedish white iron (pig iron *S*). The compositions of these two irons were as follows:

| | Pig Iron *A*. | Pig Iron *S*. |
|---|---|---|
| Total carbon, % .. .. .. .. | 3·93 | 4·0 |
| Silicon, % .. .. .. .. .. | 0·88 | 0·2 |
| Manganese, % .. .. .. .. .. | 0·30 | 0·10 |
| Sulphur .. .. .. .. .. | 0·145 | 0·005 |
| Phosphorus, % .. .. .. .. .. | 0·03 | 0·04 |

A charge of base iron and steel scrap, together with the necessary amount of alloy, was melted in a coke-fired, forced-draught furnace (except in the case of aluminium when the aluminium was added after the charge had melted). When molten, the melt was treated with a weighed amount of iron sulphide and from the larger melts four 1-2 in. dia. test-bars were top-poured, at successively lower pouring temperatures into green-sand moulds. In the case of melts of the Swedish white iron, the charges were smaller and only one test-bar was poured. In the copper series an additional group of melts was carried out to cast 3 in. dia. bars, and some small ingots were cast from a high-frequency induction-furnace melt.

A metallographic examination was made of the sulphide phases in these irons of low manganese and high sulphur contents to which varying amounts of nickel, molybdenum, copper, chromium, and aluminium have been added. In the absence of these elements manganese and iron sulphides would have been present. Nickel in amounts up to 20% gave no new sulphides; molybdenum did not influence the form of the sulphides until 2% of the element had been added. In the presence of low manganese contents, complex copper-sulphide/iron-sulphide/manganese-sulphide aggregates were obtained. Complex sulphides were found in all irons with chromium contents of between 1 and 30%; 2–7% of aluminium caused the sulphides to be present as an aluminium (manganese) sulphide. With sufficient manganese to balance normal sulphur contents, manganese sulphide was the only sulphide phase to be seen except in the case of high chromium contents.

Abnormal graphite structures were obtained in some of the irons containing nickel or copper. It is suggested that these are formed by the solidification of the iron in the white condition, followed by carbide breakdown after solidification. This process may be caused by the effect of temperature upon the reaction between manganese and iron sulphide.

## THE REMOVAL OF HYDROGEN FROM STEEL.

By Prof. J. H. Andrew, D.Sc., H. Lee, B.Eng., Ph.D., A. K. Mallik, B.Met., B.Sc., Ph.D. and A. G. Quarrell, D.Sc., F.Inst.P., F.I.M.

THIS paper, submitted by the Hair-Line Crack Sub-Committee of the Alloy Steels Research Committee, deals with a systematic investigation of the removal of hydrogen from a number of steels under isothermal conditions. It is shown that, for all steels, the rate of removal in the intermediate range is high, and that, for certain steels, there is also a peak on the removal curve corresponding to transformation in the pearlitic region. The law governing hydrogen removal in the austenitic range has been determined experimentally, and the empirical relationship supported by theoretical treatment. The effect of transformation upon the removal of hydrogen is illustrated, and the effect of specimen size is also dealt with. It is shown that small partial pressures of hydrogen surrounding the steel at 1,100° C. exert a considerable retarding effect upon the removal of hydrogen, although, as might be expected, this effect becomes quite small at 650° C.

In the presence of hydrogen, the transformation of a nickel-chromium-molybdenum steel was retarded in the range 600–450° C., but little effect was observed at lower temperatures. Comparison of the rate of diffusion of hydrogen through similar specimens of different steels showed that alloying elements have little, if any, effect upon the diffusion constant. This was confirmed in the austenitic range by the results obtained for the law of hydrogen removal. The evolution of hydrogen on cooling was followed, and it was found that the maximum rate

of evolution corresponded to the end of the thermal transformation.

The mechanical properties of a large number of steels have been determined after treatment in hydrogen and nitrogen, and it is shown that the embrittling effect of hydrogen is apparent in all cases where the hydrogen content is 1·0–3·0 c.c. /100 *g*.

In connection with the work on the removal of hydrogen, much evidence has been obtained upon the formation of hair-line cracks under various conditions. It was found that such cracks could appear in the absence of transformation stresses, and that these stresses are important mainly in that they determine the particular crack distribution which results. Although no cases were observed of internal crack formation in the absence of hydrogen, certain specimens showed no cracks in spite of a high hydrogen content. The view of the hydrogen-rich constituent put forward in a previous paper has been modified, in that it is not now considered to be necessarily austenitic ; it is realised that at the moment of crack formation the steel will be in an extremely brittle condition, owing to its hydrogen content. The exact nature of the hydrogen-rich constituent cannot be given, nor can a detailed explanation of hair-line crack formation be expected, until the much wider problem of low-alloy steels in general is more thoroughly understood.

## NEW ANNEALING PLANT FOR STEEL STRIP IN COILS AT THE WHITEHEAD IRON & STEEL CO., LTD., NEWPORT, MON.

By B. Jones, D.Sc. and I. Jenkins, M.Sc.

A NEW electric annealing plant was installed at the works of the Whitehead Iron and Steel Co., Ltd., Newport, Mon., in 1944, for the bright-annealing of cold-rolled mild steel strip in coils. The plant was designed and built by The General Electric Co., Ltd., for an output of 1,000 tons/week, and it incorporates many new features based on experience with a similar and older installation with an output of approximately 500 tons/week. Both plants consist of the vertical cylindrical or batch type furnaces, and the new installation alone represents the largest of its kind in this country.

In the general layout of the plant, the primary considerations have been maximum compactness and flexibility, with ready accessibility to all the key points of the installation. Sixteen annealing furnaces are arranged in two staggered rows on concrete plinths, set just below floor level. Six furnaces are each rated at 120 kW., and take annealing containers having internal dimensions of 36 in. dia. × 74 in. deep. The remaining 10 furnaces are of larger size, each rated at 130 kW., and take containers of 44 in. internal dia. × 74 in. deep. A raised platform of steel checker-plate is built around the furnaces, providing ready access to any part of the installation, and this platform is extended to include the recuperation and cooling pits. Each furnace is provided with two recuperation pits and six cooling pits holding eight annealing pots, in all the total number of pits thus holding 128 pots. This arrangement ensures a continuous annealing cycle.

There are three gas burners for the production of the controlled atmosphere, each having an output of 1,000 cu. ft. of burnt gas per hour. The burners are located in an alcove on the platform at the end of the line of furnaces. The annealing gas is connected to a " Mono " $CO_2$ recorder which gives a well-defined and continuous indication of the $CO_2$ content of the gas from the common gas main, this being a sufficient guide to gas composition. The recorder is situated near the gas burners, and is connected to four pipe-lines of small diameter, one to each burner for individual tests on the gas when required, and one to a common gas main from the burners in operation. The temperature-control cabinets and all the electrical switch-gear are situated at the rear of the furnace platform.

Details of the annealing cycle are given, and the causes of staining of the steel charge by unsuitable annealing gas and rolling oils are discussed. There is a close control of temperature in both the furnace and the charge. Tests have been made on the properties of low-carbon-steel strip annealed in various parts of the annealing container, and an Appendix is given on the effect of annealing temperature on the properties of heavily cold-reduced mild steel strip. It is shown that there is a range of annealing temperatures above 600° C. in which the work-hardening effects are effectively removed, good properties are obtained, and a suitable grain-size can be imparted to the steel.

## THE FORMULATION OF ANTI-CORROSIVE COMPOSITIONS FOR SHIPS' BOTTOMS AND UNDERWATER SERVICE ON STEEL.—PART II.

By F. Fancutt, F.R.I.C. and J. C. Hudson, D.Sc.

IN an earlier publication* the authors presented the results of two series of raft tests conducted at Caernarvon and Emsworth, respectively, which constituted the first stages in the systematic study of anti-corrosive paints for ships' bottoms that is being conducted by the Marine Corrosion Sub-Committee. These tests were intended to serve as a preliminary survey of the effects of pigmentation and of binder on the performance of paints used for the protection of steel immersed in sea-water. Thus, with a few exceptions, the paints tested in the Caernarvon series were all bound in the same medium, a modified phenol-formaldehyde / stand-oil varnish, differences in the pigment mixtures being the main variable, whilst at Emsworth the number of pigment mixtures was restricted to three but each was incorporated in 21 different media.

The results of this work may be summarised by the statement that, as regards pigmentation, formulations based on basic lead sulphate were found to be of particular promise, especially when aluminium or white lead was incorporated in the pigment mixture ; as regards the medium, various types of phenolic- or coumarone-resin varnish and a medium containing chlorinated rubber proved to be the best of the range of vehicles tested.

The next stage in the investigation was the study of the effects of combining the best pigment combinations with the most promising media. This the authors have endeavoured to do, and the present paper deals with the results of immersion tests in sea-water conducted on a further 127 formulated anti-corrosive paints applied to steel plates in a weathered condition. As in previous work, two coats of each anti-corrosive paint were applied to the specimens ; the painting was completed by applying a finishing coat of a formulated anti-fouling composition which was the same throughout.

* F. Fancutt and J. C. Hudson, *Journal of The Iron and Steel Institute*, 1944, No. II, p. 269 P.

The 127 paints used comprised various combinations of 8 different media with mixtures of 13 different pigments. On the basis of the results, one of the paints, pigmented with a mixture of 2 parts of basic lead sulphate and 1 part each of white lead, Burntisland red, and barytes, and bound in a modified phenol-formaldehyde/litho-oil medium is suggested for general use at this stage. Several other paints gave an even better performance in the raft tests than this paint and merit further investigation. Paints in a chlorinated-rubber medium were outstanding but certain technical difficulties need to be overcome before these can be used effectively on a practical scale.

## REPORT ON ANTI-FOULING RESEARCH, 1942–44.

By The Marine Corrosion Sub-Committee.

THIS report is an endeavour to collect together the salient features of the anti-fouling work of the Marine Corrosion Sub-Committee up to 1944. It describes the technique of exposure tests on anti-fouling compositions. Patch tests of a minimum area (12 × 8 in.) on a larger panel are employed for comparative work. The test patches are arranged at random to eliminate systematic errors. "Seasoned" panels painted with the best possible protective system are used. The assessment of fouling on the panels is carried out by examination in a sea-water tank, emphasis being laid on the kinds of organism present rather than on the quantity. Suitable dates for commencing tests are in spring for short-period tests and in autumn for long-period tests.

The sequence of settlement of fouling organisms is not a casual chain but is largely determined by the seasonal occurrence of the settling stages of each organism. Sequences may, however, develop on a toxic surface owing to gradual decrease in toxicity. Emphasis is laid on the importance of *qualitative* fouling assessments which determine the *time of breakdown* of the anti-fouling protection; quantitative measures, though valuable for some purposes, are used more as indicators of *external factors* in the environment. The seasonal settlement of fouling forms at Millport is described briefly, and is compared with conditions elsewhere in the British Isles.

The susceptibility of fouling organisms to poisons varies considerably and can be employed to determine grades of anti-fouling efficacy. The part played by bacterial slime in fouling is discussed at some length. It is not a necessary precursor of fouling, but is highly resistant and readily developed on rosin-containng coatings. It therefore tends to develop early and may influence subsequent settlements to a limited extent. This slime does not serve as a cumulative store of poison over a toxic paint.

The principal features of diatom, seaweed, and animal fouling organisms are described, particular attention being paid to such factors as season of occurrence, mode of settlement and attachment, and relative susceptibility to poisons. A brief analysis of the factors concerned in the fouling on ships' bottoms is accompanied by a summary of the results of a preliminary study of samples from 30 voyages to tropical waters.

The mode of action of anti-fouling coatings is demonstrated by an experiment giving a toxic border around a patch of anti-fouling composition. The toxicity appears to be produced by a thin layer of poisonous sea-water immediately over the paint surface. The loss of poison from the paint into this toxic layer is correlated with chemical analysis of the copper and mercury content of sea-water in which painted panels are immersed; this process is known as the leaching test. Under the experimental conditions prescribed, a rate of loss of copper of 10 $\mu$g./sq. cm./day in the laboratory test is an adequate indicator of successful anti-fouling properties. In the presence of mercury this rate of loss of copper may be lower.

The leaching technique is described and suitable methods for the estimation of copper and mercury in the leachate are presented. Preliminary studies of the effect on the laboratory leaching rate of varying the paint constituents are given. The most important factor is the resin component. In a rosin-linseed-oil varnish, replacement of the rosin by ester gum, acid or neutral synthetic resins, waxes, lanolin, bitumen or (by decreasing the rosin content) drying oil, all produce a fall in the copper leaching rate, which in a rosin-oil varnish is approximately proportional to the copper content of the paint film. Mercuric oxide also lowers the copper leaching rate from a cuprous oxide paint. The practical significance of this leaching test is at present limited to the prediction of success or failure of the anti-fouling properties of a formulation. It has only a very limited value in estimating the effective anti-fouling life, since the leaching slides are stored in laboratory tanks and lose poison less rapidly than in the sea. Methods of overcoming this difficulty are being explored and the existing technique is a useful laboratory tool in working out practical paint formulations.

Experiments on the use of organic poisons in anti-fouling compositions are described. Certain organic arsenicals, a number of organic sulphur compounds, and organomercurial derivatives offer possibilities of development, but a large number of highly toxic compounds fail as anti-fouling agents. This failure is very largely correlated with high solubility in the non-polar solvents. A description of a promising development in an aqueous cementiferous composition using organic poisons as anti-fouling agents is given. Compositions of this type open up the possibility of applying a single-coat system, firmly adherent to wet, rusty, or mill-scale-covered steel, which is adequate for anti-corrosive and anti-fouling purposes, and which may be dried out after long periods of immersion without any deleterious effect.

## INTERIM DESCRIPTIVE STATEMENT ON THE LEACHING-RATE TEST FOR SHIPS' ANTI-FOULING COMPOSITIONS.

By The Joint Technical Panel on the Leaching-Rate Test of the Marine Corrosion Sub-Committee.

THIS preliminary statement on the leaching-rate test for anti-fouling paints has been prepared in response to a demand from a section of the paint trade and is the result of work by investigators attached to the Marine Corrosion Sub-Committee of The Iron and Steel Institute and the British Iron and Steel Research Association, and by their collaborators on the Admiralty Corrosion Committee.

The test is designed to assess the value of anti-fouling compositions by measuring the rate of loss of toxic ingredients from the paint surface during immersion in

sea-water. Originally developed in the U.S.A. for application to anti-fouling compositions containing cuprous oxide as the only toxic ingredient, the test is being adapted for use on formulations containing more than one toxic constituent and in particular on those with copper and mercury, to which type most of the British proprietary compositions belong.

In conducting the test, small glass panels coated with anti-fouling compositions under test are stored in sea-water and transferred periodically into the leaching apparatus, where they are subjected to agitation (by bubbling) in a definite amount of sea-water for a standard time; the toxic substances leached into the water are then determined. It is essential that the ratio:

$$\frac{\text{Paint area} \times \text{Test duration}}{\text{Volume of sea-water used for leaching}}$$

is kept constant. The determination by colorimetric methods (using a "Spekker" photo-electric absorptiometer) of the small amounts of copper and mercury leached into the sea-water is described.

In the simplest case of anti-fouling compositions containing cuprous oxide only, it is known that a steady loss of copper of about 10 microgrammes/sq. cm./day is effective in preventing fouling. This is termed the "critical leaching rate" for copper. The critical rates for mercury, for copper and mercury in the same composition, and for other anti-fouling poisons are not yet sufficiently defined for statement, although it is known that the rate for mercury is less than half of that for copper.

The statement emphasises that, at present, exact standardisation of the test has not been achieved, nor has its precise significance in relation to service behaviour been fully determined. Further work on points requiring elucidation is being carried out in several laboratories.

# Cadmium Plate and Passivated Cadmium-Plate Coatings

**A communication from Mr. Frank Taylor**

*Dealing with the passivation of zinc and cadmium surfaces Mr. Taylor comments on Mr. Halls' article on the above subject published in our last issue. These comments, together with Mr. Halls' reply, will do much to clarify the position, but the results of the comparative tests on zinc, passivated zinc, cadmium, passivated cadmium, nickel, and tin, to which Mr. Taylor refers, would provide better guidance.*

"Now that hostilities have ceased and supplies of cadmium are a little easier, cadmium in many cases is slowly replacing zinc as a protective coating." This, in my opinion, is rather to be deplored as at least one of the modern high speed zinc solutions, namely, the ROVAL zinc solution, possesses many attributes of the cadmium solution, *i.e.*, good throwing power, low porosity, high cathode efficiency, easy control, and a rapid rate of deposition. It must be clearly realised that many of the old objections of zinc solutions are thus eliminated and providing a bright zinc or preferably a bright zinc plus passivation treatment are used, the difference in corrosion resistance between a cadmium and zinc deposit of equivalent thicknesses are negligible under normal corrosion conditions.

Too many people are prone to form an entirely erroneous conclusion of the superiority of cadmium over zinc, owing to the fact that zinc deposits from the average plating solutions are much less uniform than the corresponding cadmium ones and hence, although the average thickness is the same, the minimum thickness of zinc is far less and hence the corrosion resistance is inferior. Another point is that zinc rapidly forms a voluminous corrosion product in humid atmospheres. It must, however, be clearly realised that the corrosion resistance of any deposit is chiefly dependent upon the two following factors—

(1) The difference in potential between the deposit and base metal in the corroding medium.
(2) The solubility of the corrosion product in the corroding medium.

Thus if the formation of the corrosion product decreases the difference in potential between the deposit and the base metal and the solubility of the corrosion product in the corroding medium is very small, then the life of the deposited coating is entirely dependent on its intrinsic thickness. The above remarks apply to zinc deposits and I here again reiterate *that assuming the zinc deposit is of a high order, i.e., the porosity low, the deposit uniform, free from co-deposited impurities, and of fine grain structure, the difference in life between a cadmium and zinc coating of equivalent minimum thicknesses under normal corrosion conditions is negligible*, this difference only existing in the minds of prejudiced or ill-advised personnel.

I want to stress the above as I consider that the value of this paper would have been considerably enhanced if the plain cadmium and passivated cadmium deposits had also been compared with plain and passivated zinc deposits of equivalent minimum thicknesses. Extensive tests carried out by the writer in conjunction with various Government departments have fully established the value of passivated zinc surfaces and the results of many of these tests have been published. Other comprehensive tests have been carried out in two other directions, namely—

(1) The comparison of plain cadmium and passivated cadmium surfaces with plain zinc, passivated zinc, plain tin, passivated tin, nickel, and composite deposits under humid conditions. (K.110 test).
(2) A modified chromate passivation film for use as a base for paints whose stoving temperature is in excess of 180° F.

Unfortunately, the results of these tests, although made available to the various Government departments,

were not made public. These results showed some very interesting results as they were carried farther than the average tests and gave an interesting comparison of the value of these deposits when submitted to excessive humidity and proved the value of passivation both on cadmium and zinc, and the relative values of cadmium and zinc deposits. Unfortunately, these results cannot be published without sanction from the appropriate Government department.

The results obtained with the cadmium and passivated cadmium surfaces, however, under the salt spray and K.110 test were very similar to those obtained by the writer, although a large percentage of the blackening of the cadmium plate was no doubt due to the presence of some traces of co-deposited impurities in the cadmium deposit. This is often the case when the cadmium is plated at low current densities (10 amps per square foot and below) but in the higher pH solutions (above 12·5) using current densities in excess of 15 amps per square foot and especially in bright cadmium solutions this blackening is far less.

We all know that brass and copper sometimes require cadmium plating for various specific reasons, but I personally think that the direct plating of these metals is to be greatly deprecated and wherever possible either copper or brass should first be flashed with either nickel or tin and then cadmium plated in order to minimise the danger of the galvanic couple set up between the brass or copper and the cadmium under damp conditions. If this intermediate coating of tin or nickel is used, however, care must be taken to ensure that satisfactory adhesion is obtained between the cadmium and this intermediate deposit.

There is no doubt whatever that by passivating a cadmium or zinc surface the advantages accrued are out of all proportion to the small extra expense incurred. Passivation, however, must not be regarded as the panacea for all evils, and although it has many advantages the two following points must *always* be borne in mind, namely—

(1) Passivation is not, as many people imagine, the equivalent of several tenths of thousandths of an inch of cadmium or zinc. By this, I mean that you cannot expect a certain thickness of deposit followed by passivation to be equivalent to a thicker unpassivated deposit. *Corrosion resistance is invariably, assuming all other factors are correct* (*porosity, etc.*), *proportional to thickness of deposit.* One should understand this quite clearly. *Passivation increases corrosion resistance but must not be used as a means for decreasing initial thickness of deposit as this would be fatal.* To obtain maximum results the passivated film should be applied to the normal specified thickness of cadmium or zinc.

(2) The passivation film is very thin and soft and hence its mechanical abrasion resistance is low. Hence it is useless to go to the trouble of carefully passivating a zinc or cadmium surface, if careless mechanical handling is subsequently employed thereby removing the major part of the film and consequently greatly reducing the corrosion resistance of the finished article. *Care should be taken to ensure that the passivated film suffers the minimum of mechanical damage.*

As a final word, it is very important that the passivated film should be properly applied, as in my opinion, poor passivation is useless. In order to ensure the best results it is not only essential that the passivation solution should be carefully controlled, but that the cadmium or zinc base should be of the best possible nature. In order to ensure this, frequent periodic purification of the cadmium or zinc solution is advocated, along with the filtration of the solution, in order to ensure the best possible deposit. The time in the passivation solution is dependent upon the solution used and its age. Care should also be take to apply the "Diphenylcarbazide Spot Test" which the writer originated and is now fully described in D.T.D. 923. This ensures that the film contains sufficient hexavalent chromium (colour of spot—red to purple) and is of sufficient thickness (duration of colour). The abrasion test ensures its adhesion is correct.

In conclusion, it must be clearly realised that passivation, like any other process, has its limitations, and its application is not universal. If, however, its use is made with discrimination its worth will be unquestionable.

**Reply from Mr. E. E. Halls**

My acknowledgments and thanks are due to Mr. Frank Taylor for the detailed interest he has taken in my article, as well as for the trouble he has taken to comment upon it. From his letter, I form the opinion that, with regard to the exploitation of zinc and cadmium platings in industrial finishes, our views in the broad sense are closely the same, if not identical. Further, we are agreed that a sound technical background, systematic maintenance of finishing solutions, and chemical control of both solutions and product, are essentials in industrial finishing if success is to be achieved, if the confidence of the customer is to be retained, and if we are to avoid all kinds of nondescript finishes and finishing specifications that ultimately make any form of rationalisation impossible. I find all this particularly welcome because it is high time that industrial electroplating was properly recognised by all as a branch of metallurgy that is subject to scientific control in the factory as well as in the laboratory, in just the same way as, for example, the heat-treatment of metals. This state can only be universally established by the close co-operation of the technicians concerned, and by their establishment of processes on their merits in all senses, free from the fancies of some or the politics of others, and in spite of the ignorance of the old-time rule-of-thumb practices.

I think I have no real disagreements with Mr. Taylor's statements, but should like to deal with them briefly in order to bring out any specific matters that are naturally not catered for in his broad treatment of the subject.

Consideration of the quantity of metal available to the world under any normal conditions shows cadmium to be short in quantity and high in price compared with zinc. Any tendency to replace zinc by cadmium would accentuate this disparity, rendering cadmium a rare and precious metal, outside of the economic bounds of ordinary industrial finishing, as well as jeopardising its applications in other essential industries. Such a policy should be frowned upon nationally and internationally, and receive condemnation. In a brief way, this was covered in the first paragraph of my article, which stresses that cadmium plating should be reserved for certain purposes and exploited so that its particular merits can be used to advantage. I certainly deplore the use of cadmium as a substitute for zinc and for no other reason.

I note Mr. Taylor's commendation for " Roval " zinc, and confirm that this is well merited. I should like to add that in industrial plating shops in which chemical or metallurgical control is exercised, all the so-called and much abused disadvantages of cyanide zinc electrolytes have been studied and are susceptible to control. There are, therefore, a number of sound zinc plating plants in operation. Again, the fluorborate electrolytes for which big claims have been made in some quarters in America, are being studied in this country, and they may be able to add further to the quality of electro-zinc coatings. Therefore, consideration of properly deposited zinc coatings should precede any push to change to cadmium.

I think there are a number of other factors upon which the corrosion resistance of a deposit is dependent besides the two quoted by Mr. Taylor. These include the development of passivity by the coating, the limits or limitations of this passivity; re-acquirement of passivity after it has been lost (temporarily) by virtue of change in the conditions obtaining; mechanical stresses in the deposit by virtue of its mode of deposition; bared base metal, not only due to porosity or scratches, but by intentional operations of tapping or re-tapping, filing, etc., operations that can be condemned, yet which are commonly resorted to in engineering; bi-metallic junctions caused by other metals in the assembly, even remote from the component itself; mechanical stresses in the coating by virtue of its sacrificial corrosion, etc., etc. This, of course, is a subject in itself. At the same time, I should like to support Mr. Taylor's point by asserting that evidence has yet to be published to show the superiority of cadmium as a protective coating, quality and thickness equivalent, in any single application.

I am sorry that parallel test results could not be given for zinc and passivated zinc coatings. Space did not permit, and the real objective was to establish the attributes of the chromate passivation. There were two reasons for this, viz., firstly because the benefit of passivating cadmium is obviously not realised by many, and secondly because without passivation cadmium (other things being equal) fails before zinc. At least that is the writer's experience!

I think it would be useful to technicians and to industry as a whole if Mr. Taylor pressed for the publication of the data he mentions. There is obviously no purpose in the Services withholding it now, nor at any time if it were expressed in general terms without reference to details of the equipment involved.

The blackening of the cadmium under test was no doubt due to co-deposited impurities, and this was intentionally ignored because the same cadmium deposit was the basis of the chromatised samples. Again, this quality of cadmium, although to be deplored, is a common commercial product. Regarding the higher current density cadmiums, a warning should be given to ensure that electrode efficiencies are correct, and that the cathode efficiency is 100%. Otherwise, highly stressed deposits may result. Stressed deposits show early corrosion fatigue, this being evidenced by flaking of the deposit under corroding conditions, and consequently very poor serviceability. Some forms of this deposit stress can be studied quite effectively by X-ray diffraction methods.

With reference to cadmium coatings on copper and brass, thin coatings are hopeless, but thick ones appear to be satisfactory. I agree they should only be used when the properties of the cadmium are necessary for specific reasons, and concur with the suggestion to use an undercoat of tin to reduce the detrimental effect of the couple.

Naturally, passivation should only be adopted for its own value. Certainly, no attempt must be made to reduce the thickness of cadmium or of zinc by virtue of the passivation. On the contrary, passivation should not be applied to coatings below a certain thickness nor devoid of a certain ratio of maximum to minimum thickness. Likewise, the mechanical strength (or lack of strength) of the chromate film must be recognised; yet this serves to strengthen the campaign for more respectful handling of finished work in any case.

The proper application of the passivated film, the control of process, and chemical inspection are agreed to as essentials.

I feel gratified that Mr. Taylor, who is a recognised expert in passivity matters, has no criticism of the real facts in my article. I should now like to mention two specific applications that I consider important for cadmium, viz.

(1) For radio-frequency conductance:

(*a*) In the early days of restrictions upon the use of aluminium light alloys (*i.e.* about 1937) some designs had to be changed to steel with the minimum of redesign of the equipment as a whole. This could not be achieved with zinc plating, but could with heavy cadmium on account of the higher electrical conductivity of the cadmium.

(*b*) Other instances of cadmium plating which have enabled steel to be used in radio designs embrace those in which still greater precautions are necessary for providing conductance at radio frequencies, and in which, therefore, all junctions in chassis and the like have to be in continuous metallic contact. The ease with which cadmium can be soldered using resin cored solder, and the resin subsequently removed by means of a trichlorethylene degreaser, enables cadmium plate to be successfully employed.

(*c*) I understand that the radio industry in general prefer cadmium for domestic radio chassis fabricated from steel, not only for conductivity reasons, but for solderability, serviceability, economic production, and sales aspects. Economics can be a strong national argument for the use of cadmium in such cases.

(2) For soldering.

The electrical industry must use non-corrosive fluxes and must achieve quick soldering to give sound joints, free from any suspicion of " dry joints." This soldering often has to be done with minimum of heating to avoid damage to other joints, to plastic insulation, etc. Properly deposited cadmium fulfils this requirement, and it retains its properties of solderability in good measure after several years of storage or installation, an essential feature. Up to date, even tin coatings have not proved so good, particularly after long storage. This potentiality covers a large field in the electrical industry, especially the radio and communications industries.

Another important, although less conspicuous application concerns moving elements, especially small shafts of steel other than rust resisting grades. Zinc is often unsuitable as a coating because it "mushes," *i.e.*, it powders, and dry or lubricated collects to a mass that causes loss of efficiency and finally seizing. Nickel flakes and produces its own peculiar problems. Cadmium has, it is the writer's experience, been found to be the most serviceable solution to this problem.

Again it is stressed that these, among others, are specific applications. Yet they are important and no by means insignificant.

In response to Mr. Taylor's request, the following brief comparison of cadmium, zinc, and passivated zinc coatings can be given. They can be regarded as typical with respect to commercial finishes on small steel articles of complicated profile, with forms and holes. The coating thicknesses were about 0.0004 in. The test to which subjected was K.110, 22 wet cycles :

| No. of Cycles | Zinc | Zinc Passivated | Cadmium |
|---|---|---|---|
| 1 | General white corrosion. | Unaffected. | Darkened. |
| 2 | Increased whitening. | A few indications of slight whitening. | Dirty black patches and a few moist spots deve oped. |
| 3 | Whitening still increasing, traces of rust in bends. | Whitening along edges. | Rusting increased. |
| 22 | Heavy white corrosion and slight rusting. | Slight whitening, no rusting, much superior to impassivated zinc. | Very dark but litt'e further increase in rusting. |

These tests confirm that passivated zinc is superior to cadmium, but does not confirm that cadmium develops white growth as easily as does plain zinc. The results only in part agree with service experience and do not reveal the practical fact that cadmium *does* under certain conditions, rapidly fail. This failure, evidenced by white powdery growth, cannot be fully explained because its appearance is not universal. It occurs frequently in the hot-humid period which is experienced in this country about June. The evidence is that it does not occur with low current density cadmium deposits of high density, but that it does with rapidly deposited, less controlled coatings. This, however, needs confirmation. Also it has been suggested that organic materials such as bakelite, varnished cambric, and the like, which are invevitably present near cadmium plate when used in electrical equipment, promote the corrosion of cadmiums to white compounds by virtue of contact or proximity. This, however, has not been confirmed at all in the laboratory, and certainly is not experienced in practice in the majority of cases.

In conclusion, it is asserted that cadmium has a position of importance in industrial finishing, but its selection must be on a national basis, its availability from the quantity aspect must be regarded by all who contemplate its use, and certainly no cadmium "fashion" or complex must be allowed to develop or the price aspect will be ruined for those who legitimately need this metal.

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# Royal Commission on Awards to Inventors

By S. T. Madeley

## Rules of Procedure Governing Applications for Remuneration are Discussed

ACTING on the precedent set after the last war, the Crown has by Royal Warrant again appointed a Royal Commission on Awards to Inventors, which will have regard to, without being bound by, the principles guiding the earlier Royal Commission. A Lord Justice of Appeal, Sir Lionel Leonard Cohen, has been chosen as Chairman and the Commissioners are : Kenneth Raymond Swan, Esq., K.C. (Chairman of the Board of Trade Committee on Patents), Sir John Henry Maitland Greenly, Sir James Rae, Sir Albert George Lee, Sir William Arthur Stanier and George Macdonald Bennett, Esq., D.S.C.

The Commission has to deal with applications for remuneration, for Crown user of patents and unpatented inventions, and also under the agreement of 1946 between this country and the U.S.A. relating to Interchange of Patent Rights and Information.

There are four heads to the Royal Warrant under which Statements of Claim are made in respect of :—

1. The use of a patented invention or registered design where no terms of use have been agreed between the owner of the patent or registered design and the Government Department concerned.

2. The use of any invention or design where terms of use have been agreed or are in course of agreement between the owner of the invention or design and any Government Department.

3. The use by any Government Department or with the approval of a Government Department by any Allied Government (other than a Dominion Government and the Government of India) of any invention, design, drawing or process, not comprising a monopoly or statutory right to compensation.

4. A licence granted to the United States Government authorising use of an invention, discovery or design.

In each case when the claim has been filed, the relevant Government Department has to file an answer to the claim in question. The original claim has to be sent in duplicate to the Secretary, the Royal Commission on Awards to Inventors, Somerset House, Strand, London, W.C. 2, and a copy to the Government Department concerned. A copy of the departmental reply has to be sent to the claimant or his representative. According to the rules of procedure the claimant must first submit his claim in writing to the one or more Government Departments concerned.

If within a reasonable time no settlement is reached, this should be notified to the secretary of the Royal Commission, who will then inquire, if head 1 is involved, whether the parties are willing to refer the claim to the Commission. On this consent being given and on the claimant agreeing to accept as final the Commission's decision, settlement and determination, the secretary

will notify the parties that the Commission is willing to hear the claim.

In cases coming under heads 2, 3 or 4, on the Treasury's request the Commission will entertain the claim and the claimant will be notified by the secretary.

A month (extendable) from the date of the aforesaid notification is allowed the claimant to lodge his statement of the particulars and grounds of his claim. A month (extendable) is allowed for the Crown's reply. Within a month (extendable) the claimant may lodge an answer to the Crown's reply.

When the claim is ready for hearing, a hearing will be appointed.

A claimant may appear before the Commission personally, or by counsel, or by a solicitor, or by a patent agent.

There are four forms of Statement of Claim to be made which will now be summarised. They correspond with the four heads of the Royal Warrant. In form 1 the claimant is required to give : (1) His name and address, his description and full details of his Crown service, if any ; (2) the name and address of any solicitor or agent authorised to act for him ; (3) the subject matter of the claim, also the patent numbers and the registered design numbers ; (4) particulars of foreign patents and designs corresponding to (3) ; (5) particulars, including names and addresses of assignees, licensees or other parties interested in the patents or designs ; (6) particulars of alleged Crown user, including extent, nature and period of use, and personal service relevant to the claim ; (7) particulars of user by others than the Crown and the terms thereof ; (8) amount of compensation claimed for the user and how computed ; (9) brief details of outlay and expenses if claimed ; (10) payments already received from the Crown for use of the invention or design ; (11) brief particulars of any contract between the claimant and the Crown concerning the invention or design ; (12) nature of technical assistance supplied by Crown ; (13) particulars of economy effected by user ; (14) address of Government Department to which copy of the statement has been sent.

The claimant has to sign his agreement to acceptance as final the decision or recommendation of the Royal Commission.

In form 2, headings 1 to 7 are as in form 1 under 8, where is a request for full particulars of any agreement or proposed agreement with any Government Department ; (9) calls for a statement as to any term of agreement or proposed agreement not yet settled ; (10) requests a statement of the basis on which has been reckoned any suggested payment under (8). Under (11) particulars must be given of payments on account for user of the relevant invention or design. Heading 12 requests the address of the Government Department to which a copy of the statement has been sent.

In form 3, headings 1 to 3 are as in form 1 ; under (4) particulars with names, addresses and so on of other assignees, licensees, etc., are to be given ; Crown user and personal service rendered to the Crown by the claimant has to be particularised under (5) ; under (6) particulars are to be given of disclosure of the relevant matter in question which has led to use by the Crown or an allied Government. Exceptional utility or other special circumstances forming a basis of the claim must be set out in particulars under (7) ; remuneration (and its manner of composition) for Crown user must be stated under (8) ; and a special claim for outlay and expenses under (9) ; relevant Government contracts between claimant and Crown must be set out in brief under (10) ; technical assistance given by the Government under (11) ; details of payment already received on account for Crown user under heading 12, and under 13 the address of the Government Department to which a copy of the statement has been sent.

In form 4, headings 1, 2 and 3 are as in form 1. Under heading 4 should be stated the full terms of the licence granted to the U.S. Government under the agreements concerning the Interchange of Patent Rights and Information between the Governments of the United Kingdom and the United States ; (5) calls for particulars including names and addresses of assignees, licensees or other parties interested in the invention ; design or discovery in question ; (6) requires particulars of extent, nature and period of U.S. Government user ; user (including terms) by persons other than Government Departments under (7) ; under (8) there has to be stated the amount claimed and its basis of computation ; under (9) particulars of payments in respect of the grant of the licence which have already been received ; (10) is for brief details of claims which may have been submitted on the other three heads of the Royal Warrant. The address of the Government Department to which a copy of the completed document has been sent is to be given under (10).

Where complicated cases or those involving large amounts are concerned, the statements may need to be more detailed than the somewhat limited space on the forms will permit. The statements can then be included as schedules, numbered so that they will correspond with the headings on the forms.

A fifth form is provided for the answer by the relevant Government Department to the aforesaid claim. Under the first heading the Department has to say what it admits and what it disputes ; under (2) a statement has to be made whether the Department admits the validity of the invention or design in question and under (3) the ground of objection (shortly) if validity is disputed ; the nature, extent and period of Crown user (together with proper accounts) are given under (4). The amount of payment suggested by the Crown and its manner of computation are given under (5). Objections and further observations, including prior record and trial by or on behalf of the Crown are given under (6). The form has to be signed by an official and dated.

The Commission will not deal with claims for user by the Governments of the Dominions and of India. Such inquiries should be addressed to the Governments concerned. If there has also been user by the British Government inquiries should be made of the relevant Government Department.

As regards the heads of terms of reference, under head (1) the Commission will only deal with a claim where the claimant agrees to accept the Commission's direction as final, and provided the relevant Government Department agrees to the claim being referred to the Commission for decision.

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JOHN HARPER (MECHANITE) LTD. have re-opened their London office, and readers who find it convenient, should address their enquiries to John Harper & Co. Ltd., Chandos House, Buckingham Gate, London, S.W.1, or telephone Abbey 3184, where they can be assured of the prompt and intelligent service for which this Company is noted.

# Ductility of Metals and Alloys used in Construction-III

## The Effects of Notching under Axial and Eccentric Loads

By Dr. Georges Welter

*Ecole Polytechnique, Montreal*

*This paper records and discusses results of tests made on construction materials in order to establish a classification, with respect to their ductility, of light metals and alloys in comparison with other heavy structural materials. The effect of notching under axial and eccentric loads has also been investigated. For this purpose, standard and special notched specimens were tested under axial and eccentric loads producing single tension as well as combined tension and bending stresses.*

*Eccentricities up to ½ in. seem to have no harmful influence on standard specimens of mild steels while light alloys and brass are not ductile enough to stand these critical conditions. In specially brittle material such as cast iron there appeared to be a closer relationship between the ultimate strength and the degree of eccentric loading.*

*Similar results were obtained with conical specimens which provided a better differentiation amongst materials of different degrees of ductility.*

*Stress-strain curves of commercial metals and alloys in the "as-received" as well as in the "cold-stretched" and the "annealed" conditions, and tested under various eccentricities, give interesting information regarding the ductility of these materials. The energy absorbed during the loading process also permits a certain evaluation of the materials with regard to their strength and ductility. These diagrams show that for eccentrically loaded notched specimens, very ductile materials lose little of their properties as compared with axial loading, while less ductile and brittle materials lose rapidly their ultimate resistance and stand very small amounts of energy; these characteristics provide a useful index for the evaluation of strength and ductility. Nickel-copper and nickel-iron alloys are, in this respect, outstanding and far superior to light alloys.*

*A certain classification is given based on various indices for ductility and strength properties of notched, axially and eccentrically loaded specimens.*

### 6.—Various Indices of Ductility and Strength Properties

#### Tensile Tests

The elongation and the reduction of area are often regarded as indices permitting to measure the ductility of different materials. Taking these values from columns 5, 6 and 7 in Table IV, we obtain a classification of the materials tested, in which zinc, Monel RA and nickel occupy the highest rank for maximum elongation, and zinc, Monel RA and copper lead the field for reduction of area. The brittle materials such as cast aluminium or cast iron occupy the lowest rank in this classification. It is evident that these properties, relating exclusively to the plastic deformation of the material, are to a certain extent independent of the strength properties. The amount of energy absorbed during the test and measured by the area under the stress-strain curve, seems to be the one value which gives an indication of favourable strength-ductility relation sought for in metals. It will be noted from the results of tensile tests that a large amount of energy is required to break specimens of high resisting and plastic material. The classification obtained by using the total energy absorbed as a measure is quite different from that obtained by using the ductility index. Here the various Monel metals K, nickel and stainless steel, show the highest values (column 3, Table IV). The brittle materials such as cast aluminium and cast iron, or even wrought aluminium and magnesium alloys I and II show the lowest values.

A somewhat different classification is obtained when using the values obtained from tests on notched specimens (columns 9, 10 and 11 in Table IV). Here it is seen that the ultimate load is very high for Monel K SB, SD and SA as well as for medium steel BB, while the energy absorbed is a maximum for the Monel metals K and for Cr-Va steel.

Now taking the values for strength and energy obtained from eccentric tests (columns 13 and 14 in Table IV), we obtain a classification which does not vary appreciably from the classification obtained from the results of axial tests. Ni-Cr steel takes the place of Cr-Va steel for ultimate strength and the four Monel metals K and RA rank highest for energy absorbed.

Considering that materials are generally sought which combine a high degree of ductility with good strength properties in service, a classification which would take into consideration all properties measured during the tensile test would be useful. By using for each property a factor which varies according to the importance of the property, there may be found a common scale to evaluate the different metals. To this end, all mechanical properties of the materials tested must be compared. This may sound like measuring wine, apples and eggs with the same standard; but it should be remembered that the final figure obtained has merely a statistical value and indicates the number of merit marks that the material possesses. This method resembles somewhat

TABLE IV.—TENSILE
STATISTICAL SURVEY OF RESULTS AND CLASSIFICATION OF
MATERIAL " AS

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|

STANDARD SPECIMENS

| Material | Ultimate strength ×0·001 | Class | Yield strength ×0·001 | Class | Energy ×0·1 | Class | Total 1+2+3 | Class | Elong. ×1 | Class | Reduct. area ×1 | Class | Total 5+6 | Class | Total 6+7 | Class |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. Alum., Cast | 9·9 | 1 | 9·9 | 1 | 1·4 | 1 | 21·2 | 1 | 2·0 | 3 | 0·0 | 1 | 2·0 | 3 | 23·2 | 1 |
| 2. Alum., Wrought | 19·1 | 2 | 18·5 | 3 | 32·5 | 4 | 70·7 | 4 | 20·0 | 7 | 62·2 | 19 | 82·2 | 14 | 152·9 | 5 |
| 3. Brass | 57·6 | 9 | 48·6 | 16 | 152·0 | 10 | 258·2 | 9 | 26·0 | 14 | 33·2 | 8 | 59·2 | 9 | 317·4 | 8 |
| 4. Copper | 36·3 | 6 | 34·2 | 8 | 132·0 | 9 | 202·5 | 8 | 40·0 | 19 | 73·2 | 24 | 113·2 | 24 | 315·7 | 7 |
| 5. Cast Ir. Cr. | 20·8 | 3 | 20·8 | 4 | 6·0 | 3 | 47·6 | 2 | 1·0 | 1 | 0·0 | 1 | 1·0 | 1 | 48·6 | 2 |
| 6. Cast Ir. Ni. | 22·9 | 4 | 22·9 | 5 | 4·8 | 2 | 50·6 | 3 | 1·0 | 2 | 0·0 | 1 | 1·0 | 1 | 51·6 | 3 |
| 7. Duralumin | 65·0 | 13 | 38·9 | 12 | 195·8 | 11 | 298·8 | 10 | 25·0 | 13 | 32·0 | 7 | 57·0 | 7 | 355·8 | 10 |
| 8. Iron, Armco | 66·6 | 14 | 65·0 | 20 | 104·5 | 7 | 336·1 | 12 | 22·0 | 9 | 59·0 | 14 | 81·0 | 13 | 417·1 | 11 |
| 9. Magnesium I | 43·7 | 7 | 27·7 | 7 | 89·0 | 6 | 160·4 | 6 | 18·0 | 6 | 21·0 | 5 | 39·0 | 5 | 199·4 | 6 |
| 10. Magnesium II | 47·5 | 8 | 36·2 | 9 | 38·2 | 5 | 121·9 | 5 | 3·0 | 4 | 4·0 | 4 | 7·0 | 4 | 128·9 | 4 |
| 11. Monel RA | 86·0 | 17 | 39·4 | 13 | 355·0 | 20 | 480·4 | 19 | 50·0 | 25 | 75·0 | 25 | 125·0 | 25 | 605·4 | 20 |
| 12. Monel K SA | 125·0 | 24 | 92·5 | 23 | 530·0 | 22 | 647·5 | 21 | 20·0 | 7 | 53·4 | 13 | 73·4 | 11 | 720·9 | 21 |
| 13. Monel K SB | 172·0 | 26 | 132·0 | 26 | 800·0 | 24 | 1104·0 | 25 | 22·0 | 9 | 44·8 | 10 | 66·8 | 10 | 1170·8 | 25 |
| 14. Monel K SC | 93·5 | 20 | 57·5 | 11 | 825·0 | 25 | 956·0 | 24 | 41·5 | 21 | 62·8 | 20 | 104·3 | 20 | 1060·3 | 24 |
| 15. Monel K SD | 152·0 | 25 | 87·3 | 22 | 890·0 | 26 | 1129·3 | 26 | 29·2 | 15 | 44·4 | 9 | 73·6 | 12 | 1202·9 | 26 |
| 16. Nickel | 59·0 | 10 | 16·0 | 2 | 590·0 | 23 | 665·0 | 22 | 45·0 | 23 | 60·5 | 18 | 105·5 | 22 | 770·5 | 22 |
| 17. Steel, mild I | 64·6 | 12 | 40·6 | 14 | 249·0 | 16 | 354·2 | 14 | 41·0 | 20 | 59·5 | 15 | 100·5 | 19 | 454·7 | 15 |
| 18. " " II | 69·2 | 11 | 36·5 | 10 | 242·0 | 14 | 338·7 | 13 | 44·0 | 22 | 65·5 | 22 | 109·5 | 23 | 448·2 | 14 |
| 19. Steel, med. B | 81·2 | 16 | 50·3 | 18 | 250·0 | 17 | 331·5 | 11 | 37·0 | 18 | 67·5 | 23 | 104·5 | 21 | 436·0 | 13 |
| 20. " " BB | 128·0 | 23 | 118·0 | 25 | 280·0 | 18 | 526·0 | 20 | 14·0 | 5 | 45·1 | 11 | 59·1 | 8 | 585·1 | 19 |
| 21. " " BA | 91·5 | 19 | 56·8 | 19 | 300·0 | 19 | 448·3 | 18 | 33·0 | 16 | 60·8 | 17 | 93·3 | 16 | 541·6 | 18 |
| 22. Steel, hard C | 86·0 | 18 | 44·7 | 15 | 246·0 | 15 | 376·7 | 16 | 24·0 | 11 | 31·5 | 5 | 55·5 | 5 | 432·2 | 12 |
| 23. Steel, CA | 118·4 | 22 | 71·0 | 21 | 525·0 | 21 | 714·4 | 23 | 47·0 | 24 | 48·7 | 12 | 95·7 | 17 | 810·1 | 22 |
| 24. Steel, Ni-Cr | 99·1 | 21 | 95·2 | 24 | 230·0 | 12 | 424·3 | 17 | 24·0 | 11 | 59·4 | 15 | 83·4 | 15 | 507·7 | 17 |
| 25. Steel, Cr-VaG | 77·9 | 15 | 49·4 | 17 | 240·0 | 13 | 367·3 | 15 | 35·0 | 17 | 64·7 | 21 | 99·7 | 18 | 467·0 | 16 |
| 26. Zinc | 28·1 | 5 | 24·3 | 6 | 150·0 | 8 | 182·4 | 7 | 67·0 | 26 | 90·6 | 26 | 157·6 | 26 | 340·0 | 9 |

the one sometimes used to grade students in a class: the marks obtained in each subject by the student are multiplied by a factor of importance and the resulting total of marks for all subjects gives the rank of the student.

For the classification of the materials tested, a factor of importance for the strength properties of 1/1000 was chosen, giving a differentiation of 1 : 15 for ultimate strength and 1 : 13·2 for yielding. Considering that the energy absorbed (area under the curves) is related both to the strength and ductility properties, and consequently is of importance, a factor for this value of $\frac{1}{10}$ was chosen, giving a good differentiation of about 1 : 600 for the materials tested.

Because of the importance of the ductile properties in the final value, a factor of one was applied to the figures for the elongation and the reduction of area. These figures have a differentiation ratio of 1 : 67 for elongation and 1 : 90 for reduction of area.

The resulting classification of the materials tested, according to their tensile properties, is given in columns 16 and 17, Table IV, both for notched and eccentrically loaded specimens. Monel metal K, stainless steel, nickel and nickel-chrome steel head the list, while at the other end are found cast iron and aluminium, wrought magnesium and aluminium alloys as well as brass, zinc and copper. It will be seen from this classification that the scale for comparing these materials with regard to their merit, is very wide. A good differentiation of all materials, starting with the very brittle metals up to the high ductile alloys is possible. Between these extreme values, a scale of 1 : 48 is available, allowing a very sensitive differentiation of the materials tested.

If it should be desired to lay more stress upon the plastic flow of materials, as for instance in their production, the ductile properties could be brought out by using larger factors of importance, and the strength values could be dimmed by using smaller factors. In such a case, it is necessary to make special tests at high temperatures, in order to determine the degree of ductility of the materials at production temperatures.

TABLE V—TENSILE
STATISTICAL SURVEY OF RESULTS AND CLASSIFICATION OF
MATERIAL " COLD-STRETCHED "

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|

STANDARD SPECIMENS.

| Material | Ultimate strength × 0·001 | Class | Yield strength × 0·001 | Class | Energy × 0·1 | Class | Total 1+2+3 | Class | Elong. × 1 | Class | Reduct. area × 1 | Class | Total 5 + 6 | Class | Total 4 + 7 | Class |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1. Brass | 59·0 | 4 | 54·9 | 4 | 112·0 | 5 | 225·9 | 4 | 2·0 | 1 | 52·0 | 7 | 54·0 | 4 | 279·9 | 4 |
| 2. Copper | 38·4 | 1 | 37·1 | 1 | 92·0 | 3 | 167·5 | 3 | 30·0 | 13 | 75·0 | 13 | 105·0 | 13 | 272·5 | 3 |
| 3. Duralumin | 74·8 | 6 | 68·7 | 5 | 126·0 | 7 | 269·5 | 6 | 15·0 | 6 | 34·0 | 4 | 49·0 | 3 | 318·5 | 6 |
| 4. Iron, Armco | 68·8 | 5 | 68·8 | 6 | 97·0 | 4 | 234·6 | 5 | 17·0 | 8 | 55·0 | 9 | 72·0 | 9 | 306·6 | 5 |
| 5. Magnesium I | 46·8 | 3 | 44·6 | 3 | 53·0 | 2 | 144·4 | 1 | 9·0 | 2 | 15·0 | 1 | 24·0 | 1 | 168·4 | 1 |
| 6. Magnesium II | 46·1 | 2 | 42·2 | 2 | 66·0 | 1 | 154·3 | 2 | 13·0 | 5 | 17·0 | 2 | 30·0 | 2 | 184·3 | 2 |
| 7. Monel RA | 118·0 | 12 | 118·0 | 12 | 200·0 | 12 | 436·0 | 12 | 10·0 | 4 | 54·0 | 8 | 64·0 | 7 | 500·0 | 12 |
| 8. Mild Steel II | 81·3 | 7 | 81·3 | 8 | 115·0 | 6 | 277·6 | 7 | 17·0 | 8 | 56·0 | 10 | 73·0 | 10 | 350·6 | 7 |
| 9. Med. Steel B | 93·0 | 9 | 93·0 | 9 | 164·0 | 10 | 350·0 | 10 | 22·0 | 11 | 66·2 | 11 | 88·2 | 11 | 438·2 | 11 |
| 10. Med. Steel BA | 114·2 | 11 | 114·2 | 11 | 132·2 | 8 | 360·4 | 11 | 9·0 | 2 | 45·0 | 6 | 54·0 | 4 | 414·4 | 8 |
| 11. Hard Steel C | 100·9 | 10 | 100·9 | 10 | 148·0 | 9 | 349·8 | 9 | 18·0 | 10 | 41·0 | 5 | 69·0 | 8 | 418·8 | 9 |
| 12. Steel, Stahl. | 139·1 | 13 | 137·0 | 13 | 374·0 | 13 | 650·1 | 13 | 23·0 | 12 | 32·5 | 3 | 55·5 | 6 | 705·6 | 13 |
| 13. Nickel | 87·5 | 8 | 78·5 | 7 | 175·0 | 11 | 341·0 | 8 | 16·0 | 7 | 68·8 | 12 | 84·8 | 12 | 425·8 | 10 |
| 14. Alum., Wrought | 12·9 | 1 | 4·9 | 1 | 57·9 | 1 | 75·7 | 1 | 5·2·0 | 5 | 77·8 | 5 | 129·8 | 5 | 205·5 | 1 |
| 15. Brass | 53·· | 2 | 27·1 | 2 | 240·0 | 4 | 320·5 | 3 | 44·0 | 4 | 57·9 | 3 | 101·9 | 4 | 422·4 | 3 |
| 16. Iron, Armco | 56·7 | 3 | 44·7 | 3 | 138·0 | 2 | 249·4 | 2 | 31·0 | 3 | 59·3 | 4 | 90·3 | 3 | 339·7 | 2 |
| 17. Med. Steel BB | 99·9 | 4 | 63·4 | 4 | 274·0 | 5 | 437·3 | 4 | 28·0 | 2 | 53·3 | 2 | 81·3 | 2 | 518·6 | 4 |
| 18. Steel EA | 128·6 | 5 | 104·8 | 5 | 232·5 | 3 | 465·9 | 5 | 18·0 | 1 | 47·0 | 1 | 65·0 | 1 | 530·9 | 5 |

PROPERTIES
MATERIALS ACCORDING TO VARYING "MERIT POINTS."
RECEIVED."

| | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 |
|---|---|---|---|---|---|---|---|---|---|
| | NOTCHED SPECIMENS | | | | ECCENTRIC LOADED SPECIMENS | | | | |
| | Ultimate strength ×0·001 | Class | Energy × 0·1 | Class | Total 9 + 10 | Class | Total 8 + 11 | Class | Ultimate strength ×0·001 | Class | Energy × 0·1 | Class | Total 13 + 14 | Class | Grand Total 12 + 15 | Class | Rank of materials Gen. Classif. |
| 1. | 11·42 | 1 | 2·4 | 1 | 13·8 | 1 | 37·0 | 1 | 13·4 | 1 | 3·6 | 1 | 17·0 | 1 | 54·0 | 1 | 1. Alum., Cast |
| 2. | 29·8 | 4 | 10·8 | 4 | 41·6 | 3 | 194·5 | 4 | 79·9 | 5 | 37·0 | 8 | 116·9 | 6 | 311·4 | 5 | 2. Cast Ir. Cr. |
| 3. | 72·2 | 9 | 25·2 | 9 | 97·4 | 9 | 414·8 | 9 | 84·8 | 7 | 33·6 | 7 | 118·4 | 7 | 533·2 | 7 | 3. Cast Ir. Ni. |
| 4. | 56·0 | 8 | 21·6 | 8 | 77·6 | 8 | 393·3 | 7 | 161·5 | 11 | 68·4 | 11 | 229·9 | 11 | 623·2 | 9 | 4. Magnesium II |
| 5. | 20·0 | 2 | 4·8 | 2 | 24·8 | 2 | 73·4 | 2 | 19·9 | 2 | 4·8 | 2 | 24·7 | 2 | 98·1 | 2 | 5. Alum., Wrought |
| 6. | 21·7 | 3 | 6·0 | 3 | 67·7 | 6 | 119·3 | 3 | 20·9 | 3 | 4·8 | 2 | 25·7 | 3 | 145·0 | 3 | 6. Magnesium I |
| 7. | 82·2 | 10 | 38·4 | 13 | 120·6 | 10 | 476·4 | 10 | 130·0 | 8 | 4·8 | 2 | 178·0 | 8 | 654·4 | 10 | 7. Brass |
| 8. | 100·9 | 14 | 43·2 | 14 | 144·1 | 13 | 561·2 | 11 | 226·1 | 12 | 94·8 | 12 | 320·9 | 12 | 882·1 | 12 | 8. Zinc |
| 9. | 47·1 | 5 | 16·9 | 6 | 64·0 | 4 | 263·4 | 6 | 84·1 | 6 | 31·2 | 6 | 115·3 | 5 | 378·7 | 6 | 9. Copper |
| 10. | 50·7 | 7 | 18·0 | 7 | 68·1 | 7 | 197·6 | 5 | 45·6 | 4 | 8·4 | 5 | 54·0 | 4 | 251·6 | 4 | 10. Duralumin |
| 11. | 117·2 | 17 | 101·0 | 19 | 218·2 | 18 | 823·6 | 17 | 349·2 | 19 | 330·2 | 20 | 688·4 | 19 | 1512·0 | 17 | 11. Steel, hard |
| 12. | 190·5 | 22 | 174·0 | 21 | 364·5 | 21 | 1085·4 | 21 | 424·0 | 22 | 361·5 | 22 | 785·5 | 22 | 1870·9 | 21 | 12. Iron, Armco |
| 13. | 226·0 | 24 | 237·0 | 24 | 463·0 | 24 | 1633·8 | 24 | 469·0 | 23 | 401·0 | 23 | 870·0 | 25 | 2503·8 | 23 | 13. Steel, mild II |
| 14. | 110·0 | 15 | 114·0 | 20 | 224·0 | 19 | 1244·3 | 22 | 340·0 | 17 | 351·0 | 21 | 691·0 | 20 | 1975·3 | 22 | 14. Steel, med. BA |
| 15. | 194·0 | 23 | 180·0 | 22 | 374·0 | 22 | 1576·9 | 23 | 523·0 | 24 | 498·0 | 24 | 1021·0 | 24 | 2597·9 | 24 | 15. Steel, med. B |
| 16. | 82·5 | 11 | 99·0 | 18 | 181·5 | 15 | 952·0 | 19 | 258·2 | 14 | 336·0 | 19 | 594·2 | 17 | 1546·2 | 19 | 16. Steel, med. BB |
| 17. | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | 17. Monel RA |
| 18. | 84·5 | 12 | 57·8 | 15 | 142·3 | 12 | 590·5 | 13 | 253·5 | 13 | 153·4 | 13 | 406·9 | 13 | 997·4 | 13 | 18. Steel, Ni-Cr |
| 19. | 116·3 | 16 | 75·9 | 17 | 193·2 | 16 | 629·2 | 14 | 341·8 | 18 | 214·6 | 16 | 556·4 | 16 | 1185·6 | 15 | 19. Nickel |
| 20. | 188·5 | 21 | 37·2 | 11 | 225·7 | 20 | 810·8 | 16 | 295·6 | 15 | 171·6 | 14 | 467·2 | 14 | 1278·0 | 16 | 20. Steel, Stainl. |
| 21. | 127·1 | 18 | 75·6 | 16 | 202·7 | 17 | 644·3 | 15 | 317·0 | 16 | 185·8 | 15 | 502·8 | 15 | 1147·1 | 14 | 21. Monel K SA |
| 22 | 99·4 | 13 | 37·2 | 11 | 136·6 | 11 | 568·8 | 12 | 132·7 | 9 | 54·0 | 10 | 186·7 | 10 | 755·5 | 11 | 22. Monel K SC |
| 23. | 145·8 | 19 | 25·2 | 9 | 171·0 | 14 | 981·1 | 20 | 414·1 | 21 | 290·4 | 18 | 704·5 | 21 | 1685·6 | 20 | 23. Monel K SB |
| 24. | 162·5 | 20 | 216·0 | 23 | 378·5 | 23 | 885·2 | 18 | 411·7 | 20 | 235·4 | 17 | 647·1 | 18 | 1533·3 | 18 | 24. Monel K SD |
| 25. | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | — | |
| 26. | 48·1 | 6 | 16·7 | 5 | 64·8 | 5 | 404·8 | 8 | 133·0 | 10 | 50·4 | 9 | 183·4 | 9 | 588·2 | 8 | |

The classification resulting from tension tests on cold-stretched and annealed materials is given in Table V; it also provides a clear differentiation between the strength and ductility characteristics of the materials under consideration

## Conclusions

1. Universal joints, used in conjunction with cylindrical blocks provided with eccentric threaded holes at $\frac{1}{8}$ in., $\frac{1}{4}$ in. and $\frac{1}{2}$ in. off centre, produce combined tension and bending stresses in a specimen.

2. With standard specimens of steel loaded under $\frac{1}{4}$ in. and $\frac{1}{2}$ in. eccentricity, up to the ultimate strength, the initial eccentricity disappears gradually in the plastic range; owing to the self-alignment of ductile material, the load finally becomes nearly axial again. Loading mild or even medium steels under an eccentricity up to $\frac{1}{2}$ in. does not affect the breaking strength as compared with axial loading. However, brass and light alloys (such as duralumin and magnesium alloys) are not ductile enough to stand these severe testing conditions. With brittle materials such as cast iron, there is a clear relationship between the ultimate load and the degree of eccentric loading, so that for $\frac{1}{2}$ in. eccentricity the ultimate strength is only a fraction of the strength under axial loading.

3. Conical specimens, with a much smaller volume of plastic flow than standard specimens, showed, under $\frac{1}{2}$ in. eccentricity, no particular weakness. The total energy necessary to break conical specimens of steel, brass or duralumin was even higher under eccentric than under axial loading Conical specimens of magnesium alloys and cast iron, however, proved very weak under eccentric loading. Furthermore, conical specimens taken from cold-stretched mild steel lost, under $\frac{1}{2}$ in. eccentricity, only a small percentage of the ultimate strength they had shown under axial loading.

4. In order to compare commercial metals and alloys with regard to their ductility and strength properties, stress-strain curves were secured of materials in the "as received" condition and were compared to those obtained for the same materials in the cold-stretched and the annealed conditions. From these results it was found that the nickel alloys occupy a fairly good place among the materials tested. A sharp notch provided in

PROPERTIES.
MATERIALS ACCORDING TO VARYING "MERIT POINTS."
AND "ANNEALED."

| | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 |
|---|---|---|---|---|---|---|---|---|---|
| | NOTCHED SPECIMENS | | | | ECCENTRIC LOADED SPECIMENS | | | | |
| | Ultimate strength ×0·001 | Class | Energy × 0·1 | Class | Total 9 + 10 | Class | Total 8 + 11 | Class | Ultimate strength ×0·001 | Class | Energy × 0·1 | Class | Total 13+14 | Class | Grand total 12+15 | Class | Rank of materials Gen. Classif. |
| 1. | 82·2 | 4 | 37·2 | 4 | 119·4 | 4 | 399·3 | 4 | 115·0 | 3 | 37·2 | 3 | 152·2 | 3 | 551·5 | 3 | 1. Magnesium II |
| 2. | 59·4 | 2 | 19·2 | 2 | 78·6 | 2 | 351·1 | 3 | 171·9 | 6 | 70·8 | 6 | 242·7 | 6 | 593·8 | 4 | 2. Magnesium I |
| 3. | 101·9 | 5 | 50·4 | 6 | 152·3 | 5 | 470·8 | 6 | 147·3 | 4 | 63·6 | 4 | 210·9 | 4 | 681·7 | 5 | 3. Brass |
| 4. | 110·0 | 6 | 50·4 | 6 | 160·4 | 6 | 467·0 | 5 | 202·1 | 7 | 86·4 | 7 | 288·5 | 7 | 755·5 | 6 | 4. Copper |
| 5. | 60·0 | 3 | 20·4 | 3 | 80·4 | 3 | 248·8 | 1 | 99·6 | 2 | 33·0 | 2 | 133·2 | 2 | 382·0 | 1 | 5. Duralumin |
| 6. | 56·9 | 1 | 18·0 | 1 | 74·9 | 1 | 259·2 | 2 | 94·5 | 1 | 30·0 | 1 | 124·5 | 1 | 383·7 | 2 | 6. Iron, Armco |
| 7. | 157·0 | 12 | 48·0 | 5 | 205·0 | 8 | 705·0 | 12 | 460·6 | 13 | 137·8 | 9 | 598·4 | 11 | 1303·4 | 11 | 7. Steel, hard C |
| 8. | 118·0 | 7 | 54·0 | 8 | 172·0 | 7 | 522·6 | 7 | 250·1 | 9 | 142·8 | 10 | 392·9 | 9 | 925·5 | 8 | 8. Steel, mild II |
| 9. | 136·0 | 9 | 79·2 | 9 | 217·2 | 10 | 655·4 | 11 | 384·6 | 11 | 250·6 | 11 | 635·4 | 12 | 1290·8 | 10 | 9. Steel, med. BA |
| 10. | 151·9 | 11 | 81·6 | 11 | 233·5 | 12 | 647·9 | 10 | 244·2 | 8 | 126·0 | 8 | 370·2 | 8 | 1018·1 | 9 | 10. Steel, med. B. |
| 11. | 140·5 | 10 | 64·8 | 10 | 205·3 | 9 | 624·1 | 8 | 166·6 | 5 | 67·2 | 5 | 233·8 | 5 | 857·9 | 7 | 11. Monel RA |
| 12. | 202·0 | 13 | 134·4 | 13 | 336·4 | 13 | 1041·0 | 13 | 314·5 | 10 | 518·5 | 12 | 833·0 | 10 | 1874·0 | 13 | 12. Nickel |
| 13. | 135·0 | 8 | 85·5 | 12 | 220·5 | 11 | 546·3 | 9 | 408·7 | 12 | 707·2 | 13 | 1115·9 | 13 | 1762·2 | 12 | 13. Steel, Stainl. |
| 14. | 17·6 | 1 | 13·2 | 1 | 30·5 | 1 | 246·3 | 1 | 52·2 | 1 | 44·4 | 2 | 96·6 | 1 | 332·9 | 1 | 1. Aluminium |
| 15. | 65·8 | 2 | 21·6 | 2 | 87·4 | 2 | 509·8 | 3 | 116·4 | 2 | 38·4 | 1 | 154·8 | 2 | 664·6 | 2 | 2. Brass |
| 16. | 66·3 | 3 | 40·8 | 3 | 113·1 | 3 | 452·8 | 2 | 203·8 | 3 | 144·0 | 3 | 347·8 | 3 | 800·6 | 3 | 3. Iron, Armco |
| 17. | 148·2 | 4 | 96·0 | 4 | 240·2 | 4 | 762·8 | 4 | 394·9 | 5 | 295·8 | 5 | 689·7 | 5 | 1452·5 | 4 | 4. Steel, med. BB |
| 18. | 218·0 | 5 | 190·0 | 5 | 408·0 | 5 | 938·9 | 5 | 355·4 | 4 | 231·6 | 4 | 587·0 | 4 | 1525·9 | 5 | 5. Steel, Cr.-Ni |

the specimen prevents the flow of material during loading, and an increase of 20% to 40% in the ultimate strength results for most materials except cast alloys and magnesium alloys, whose strength is not or very little increased. With pure metals, increases of 50% to 72% have been measured. This notching effect takes place just the same with materials that have been subjected to the severe process of cold-stretching.

5. Interesting results have been obtained by testing notched specimens under loads applied with eccentricities of $\frac{1}{8}$ in., $\frac{1}{4}$ in. and $\frac{1}{2}$ in. While the stress-strain curves of ductile materials remain nearly unchanged under such loading conditions, less ductile materials behave quite differently. They give very characteristic stress-strain curves showing not only rapidly decreasing ultimate loads, but also sharply decreasing absorbed energy with increasing eccentricities. The original curves, reproduced in Figs. 14, 14a and 16 provide very characteristic indices of the strength and ductility of the materials tested. Especially those showing equal or increasing resistance or energy at $\frac{1}{2}$ in. eccentricity are of particular interest. Copper-nickel and iron-nickel alloys possess these properties to a high degree and are, in this respect, greatly superior to light alloys. Moreover, cold-stretched and annealed materials behave, in this respect, in a very significant manner.

6. A classification according to their ductility and strength properties is given in Table IV and V of notched specimens axially and eccentrically loaded in tension. It appears from those figures that, for resistance in tension, Monel metal K, stainless steel, nickel and nickel-chrome steels lead all other materials tested, including nickel; the same materials occupy again predominant positions in this classification for resistance in torsion. Brittle materials and light alloys occupy inferior positions in the classification for resistance in tension as well as in torsion.

# Correspondence

## Protection of Zinc Surfaces by Phosphating

The Editor, METALLURGIA.

Dear Sir,

I read with great interest the brief article on New American Methods of " Protection of Zinc Surfaces by Phosphating " in your October issue of METALLURGIA.

G. W. Jernstedt is a personal friend of mine and on this account I have been fortunate enough to have had the opportunity of testing out the efficacy of his pre-activating salt for zinc surfaces and can personally vouch that his claims are more than substantiated. As a matter of fact we are both carrying out comparative tests on the value of a pre-activated and phosphated zinc surface as compared with a chromate passivated zinc surface. Up to date these tests have not been completed, but for general purposes the following applies—

(1) The chromate passivated zinc surface is superior where maximum corrosion resistance is required (*i.e.*, where no subsequent finish is employed).

(2) The pre-activated phosphated zinc surface is superior where maximum paint adhesion is required (*i.e.*, where subsequent finish is applied).

The position on phosphating in this country is also similar to that in the States. Before about 1936 the Pyrene Company, owing to its Parker patents, virtually held a monopoly on all the phosphating processes, with the exception of coslettising, carried out in this country. Owing to the cancellation of the agreement, however, between the American Chemical Paint Co. and the Parker Rustproofing Co. in America, the I.C.I. began to operate the granodising processes of A.C.P. and are now the Parker's biggest competitors in this country. Various other phosphate processes which, in the writer's opinion, are equally, if not more, effective are also in use in this country. With the expiration of some of the basic patents, undoubtedly more phosphate processes will tend to be commercialised. Two of the most notable of these are Rovaline of International Corrodeless, Ltd., Enfield, and Walterisation of Walterisation, Ltd., Croydon.

One important fact has, however, been brought to the fore in the past 12–18 months, that, although phosphating has come to stay, unless the present high prices of the proprietary materials are considerably reduced many of the larger manufacturing concerns will tend to develop and use their own specific solutions. Hence, within the next two to five years a rather interesting situation should develop in this country, somewhat similar to that now developing in the States. The days of high priced proprietary salts in the metal finishing trade are now quickly departing and whereas during the war years price did not matter, in the coming days of competition processing costs will be of major importance, and these *must* be reduced to a minimum if firms are to survive. In addition, the present policy appears to be a prevention of monopolies in any trade and this should further stimulate competition and a general reduction in prices.

I trust the above remarks will be of some interest to your readers.

Bush Hill Park,
Enfield, Middlesex.
28th October, 1946.

Yours faithfully,
FRANK TAYLOR.

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MARSHALL RICHARDS MACHINE CO. LTD., the wire drawing division of Marshall, Sons & Co. Ltd., Gainsborough, have taken over a two-acre factory in Crook, Co. Durham, which was officially opened early this month by Mr. J. D. Murray, M.P. Following the opening ceremony Mr. Murray, members of the Crook and Willington U.D. Development Board, the directors of Marshall Richards Machine Co. Ltd., and the workpeople gathered in the work's canteen, where the meeting was addressed by Mr. Murray, Mr. George Wood (chairman of the Company), Mr. Ashley S. Ward (chairman of Marshall, Sons & Co. Ltd., Gainsborough), Mr. George Bryden (managing director), Mr. S. H. Richards (technical director), and Councillor W. Bourne (chairman of the Crook Development Board).

The factory is a very modern one and was built by the Admiralty three years ago. Marshall Richards will concentrate their production in Crook, and it is their intention to employ an increasing number of workmen in the district.

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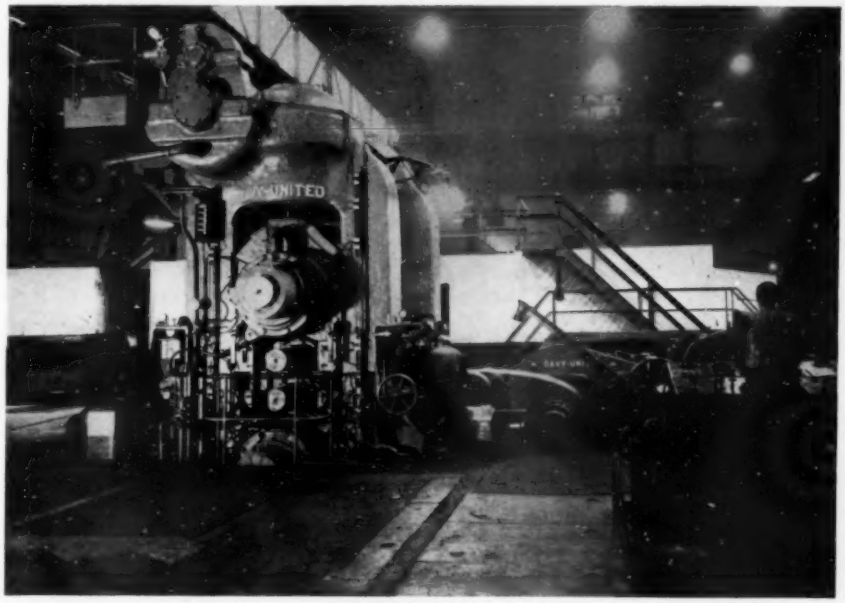
WE have learned with regret of the sudden death of Mr. H. Toplis, who has represented Kasenit, Ltd. in the Midlands for many years, where he was a well-known figure, more particularly in the Birmingham district. He was an expert on heat-treatment and his experience of the problems associated with heat-treatment processes was always available to his many friends, who will certainly miss having his advice and help.

# The New 80-in. Cold Reduction Reversing Strip Mill

## of John Summers & Sons, Ltd.

*Cold-rolling pickled, hot-rolled steel strip develops certain qualities and properties in the material, such as dimensional accuracy, surface finish, and temper or hardness, and the product is applied to numerous uses involving stamping, drawing, and other cold-forming operations. Increased facilities for cold rolling at the Shotton works of John Summers & Sons, Ltd. are provided by a new mill which is briefly described. Reference is made to the development of these works, to the products of the Company, and to future trends in the application of sheet steel.*

A FOUR-HIGH reversing mill has been installed and recently put in operation at the Hawarden Bridge Steelworks, Shotton, of John Summers & Sons, Ltd. It is designed to cold reduce hot-rolled pickled strip up to a maximum width of 74 in. and is equipped with tension reels fore and aft with a cone type uncoiler and pinch roll feeder on the entry side. A maximum coil weight of 10 tons can be handled and the mill will be capable of rolling approximately 1,500 to 2,000 tons of strip per week.

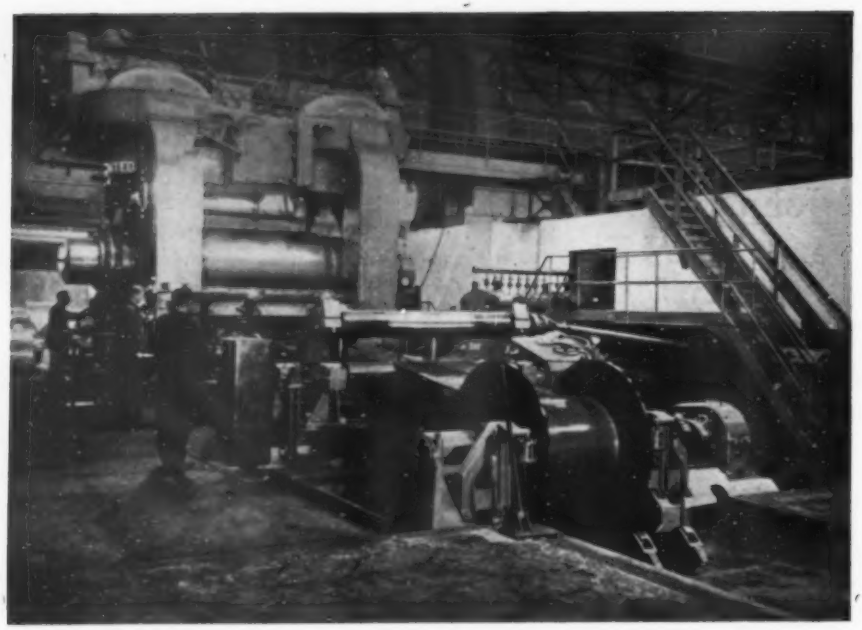


**A side view of the 80-in. cold reduction reversing mill at the Shotton works.**

In a single stand reversing unit of this type the coil is first placed on the uncoiling reel, the free end of the strip is uncoiled sufficient to pass through the rolls and attach to the opposite reel. Tension is applied through the reels sufficient to draw the strip tightly over the levelling rolls mounted on each side of the mill and the rolls are forced together by screwdowns until the steel is compressed to the reduction required for the first pass. The reels and rolls are then started and the strip passes through the mill rolls until the inner end is about to leave the reel. The rolls are then adjusted to the reduction for the second pass, and the mill is reversed, the procedure being repeated for the three or five passes required to bring the strip to the desired gauge. Usually three passes suffice but five passes may be necessary for the lighter finished gauges. The speed range of the present mill is 500 to 1,000 ft. per minute.

This installation forms part of a scheme of development planned in 1936 which included the installation of a strip mill with modern finishing equipment. Following the visit of a delegation from John Summers to America, where vast strides had been made in sheet steel production, an order was placed for a strip mill with the Mesta Machine Company and the first strip was rolled from this mill in November, 1939. The foresight displayed by the directors of this Company enabled the strip mill to make a very valuable contribution to the requirements of the Armed Forces in the recent war, but the further developments planned had to be shelved. In keeping with the progressiveness of this Company, however, there has been no unnecessary delay in carrying out the original objective.

As will be noted in the accompanying illustrations, the new mill is of heavy and rigid construction. The housings are of cast steel and each weighs about 75 tons. The working rolls which are forged steel, are 20¼ in. diameter and rotate in Timken bearings. The backing rolls, which are alloy steel castings, are 53 in. diameter and rotate in Morgoil bearings carried in heavy cast steel chocks. Pressure is maintained between the top work roll and the top backing roll by hydraulic cylinders mounted in the lower work roll chocks, the rams of these cylinders thrusting against the upper work roll chocks. Pressure is also maintained between the top backing chocks and the mill screws by a similar arrangement of hydraulic cylinders and rams in the lower backing chocks. The



**End view of 80-in. cold reduction reversing mill showing one of the tension reels.**

design is such that all roll assemblies are withdrawable from the open side of the mill.

The screw-down gear, in which the screw is 18 in. diameter is operated by two 50 h.p. motors which can be used independently or can be used as one unit through the medium of a magnetic clutch coupling the two motor spindles together. The work rolls are driven, through short universal spindles and a pinion stand, directly by a 2,500 h.p. 93/186 r.p.m. D.C. motor.

The two reels are of the collapsing type each driven by two 500 h.p. 200/675 r.p.m. D.C. motors in tandem through a 3·86/1 reduction gear. The loads imposed by the strip tension on the reels are carried directly to the mill housing. All drives are designed to keep the inertia to a minimum.

The mill motors are supplied with power from a motor generator set comprising a 3,900 h.p. 500 r.p.m. synchronous motor taking current at 6·6 Kv., driving a 2,200 Kw. 600 v. main generator and two reel booster generators of 456 Kw. ± 338 v. The main mill control is on the "Ward Leonard" system. Preset and accurate control of strip tension is ensured through the use of "Metadyne" control.

Very comprehensive lubrication equipment has been provided—the pinion stand and the reel gear reduction units have independent oil circulating sytems each fitted with duplicate pumps. Should one of these pumps fail, a second pump is automatically brought into commission and this is again protected by an alarm system. The back-up roll bearings are supplied from our main oil circulating system with protective alarms. All grease lubricated points are served by a "Farval" automatic system.

All rectilinear motions on the complete mill are actuated through the medium of hydraulic or compressed air cylinders. The hydraulic power is supplied at 750 lbs./in. from a self-contained pumping and accumulator set in the cellar of the mill. The actual operating control is from two desks either of which can be selected by the operator through the medium of a selector switch. Each desk carries a full complement of instruments necessary for the operation of the mill.

The design of the complete mill was carried out by the manufacturers in full collaboration with the staff of John Summers & Sons, Ltd. The mechanical parts of the mill were made by Davy and United Engineering Co. Ltd., and the main electrical gear by Metropolitan-Vickers Electrical Co. Ltd.

## Facilities for Sheet Production at Shotton

The installation of this new mill is an indication of the progressiveness of this firm, but since the stock used for cold rolling sheets is hot-rolled strip it will be of interest to review briefly the history of John Summers & Sons and to discuss the developments which have raised them to a position of eminence in the production of steel sheet.

Founded in 1850 by Mr. John Summers when he opened a small shop in Stalybridge to make clog irons, he subsequently bought a nail-making machine and the following year began to manufacture cut nails. The business proved so successful that Mr. Summers decided to make his own raw materials for manufacturing nails, and, in 1860, puddling furnaces, a steam hammer, and the first sheet rolling mill were installed at Stalybridge. After the death of the founder in 1876, the business was successfully carried on by three of his elder sons. In 1889 one of the younger sons, Mr. Henry Hall Summers, joined the firm, and on the death of his eldest brother he succeeded to the Chairmanship. He soon started to evolve schemes for the improvement and enlargement of the undertaking. New mills were installed at Stalybridge, iron sheets gave place to steel sheets from steel bars shipped from America, and a galvanising plant was installed for making galvanised steel corrugated roofing sheets. The first galvanised sheet from these works was produced in 1894.

It was apparent to Mr. Henry Summers that there would be an ever-increasing demand, not only in this country, but in world markets, for galvanised corrugated roofing sheets, and in 1895 he bought land near Chester, on which the Hawarden Bridge Steelworks now stands, to provide facilities for the development he contemplated. The site was chosen because of its close proximity to Liverpool and its port facilities. Other advantages included the availability of coal, a plentiful supply of water, and abundant space for expansion. In 1896 the new Shotton plant started work. It comprised six sheet mills driven by a steam engine, with galvanising and finishing equipment. By 1902 thirty mills had been

installed; in addition, No. 1 steel plant, comprising ten open-hearth furnaces of 60-ton capacity, and a bar mill had been erected and put into operation, in order to supply the greater portion of the sheet bar requirements, which until then had been imported.

Continued expansion of the galvanised sheet trade resulted in the erection of a new sheet producing plant, which was commenced in 1911 ; soon after its completion the war of 1914-1918 broke and the Shotton plant was able to play an important role in the supply of steel sheets for war purposes. During the war, and at the request of the Government, the steel making capacity was increased in 1917 by the erection of No. 2 steel plant and bar mill. This shop was equipped with eight open-hearth furnaces of 70-ton capacity, and commenced operations less than twelve months after cutting of the first sod.

**An aerial view of the steelworks at Shotton.**

Shortly after the close of that war, the works of the Wolverhampton Corrugated Iron Company at Ellesmere Port were acquired. These works had a production of steel sheets of about 2,000 tons per week. To ensure an adequate supply of coal and pig iron the Shelton Iron, Steel, and Coal Company at Stoke-on-Trent was also acquired ; in addition to mining coal and making pig iron, this Company manufactures rails, billets, steel joists, and other sections.

By 1928 it became clear that the export market, upon which Shotton so largely depended, was rapidly falling away, owing to the installation of sheet mills in many of the countries which had up to that time bought from the Shotton works, and a new market had to be found and developed. About this time the motor car industry was expanding rapidly, and its demand for sheets was increasing, the production of motor car body sheets was therefore decided upon and a small cold-rolling plant, the invention of a Polish engineer, was installed. The sole purpose of this plant was to meet the more difficult requirements in steel sheet which the motor car trade was demanding.

During the years between 1930 and 1936 America had been making great strides in the deve.opment of her sheet steel industry, her leading steel firms were installing strip mills with modern finishing equipment. A delegation from Shotton went to America to investigate these developments, and to see whether it would be prudent to install a strip mill at Shotton, and so keep the Company's products in the forefront of modern development. On the return of the delegation it was decided to place an order for a strip mill with the Mesta Machine Company of Pittsburg. The first strip from this mill was rolled in November, 1939, fortunately in time to make a most valuable contribution to the requirements of the Armed Forces in the recent war.

While the original area of these works in 1896 occupied six acres, continuous expansion has increased the area to 250 acres, 51 acres of which comprise covered buildings. It is noteworthy also that 760 acres are available for further extensions, already part of a scheme upon which a favourable decision has been made. Some figures taken at random will indicate the size of this undertaking. The hot strip mill bay is 1,487 ft. in length. There are 45 miles of works railways and 4,900–5,100 wagons are handled per week. Coal consumption varies between 5,700 and 6,300 tons per week ; consumption of electricity is $1\frac{3}{4}$ to 2 million units per week ; and water 51 to 59 million gallons per week. The total number of employees is 5,900–6,300.

*Melting Shops.* Nos. 1 and 2 steelworks are in operation, the former, as previously mentioned, having 9–60-ton, and the latter 8–70-ton basic open-hearth furnaces. With one exception all furnaces are fired with produced gas. The exception is one of the 70-ton furnaces which is being fired with fuel oil. This is in the nature of an experiment and has not been carried out long enough to present authentic results, but the general opinion is that fuel oil offers many advantages in providing a hotter and more regular flame. The use of oil fuel in open-hearth furnaces obviously needs modifications in design to obtain the best results, but whether its use effects economy in fuel costs or, if more economical than coal, whether it would be policy to use it, are questions not easily answered. It is noteworthy that the ingot production of these shops is at the rate of 8,000 to 10,000 tons per week, and the indications are that the production this year will exceed last year's production by a considerable margin.

*Slab Mill.* The rolling of the slab is the first step in the rolling of steel strip and for this purpose a 40-in. modern reversing type slab mill is installed. This mill is driven by a 3-cyl. simple steam engine operating at a

pressure of 120 lbs. per sq. in. The cylinders have a 40 in. bore by 54 in. stroke. The rolls are 38 in. diameter and the capacity of the mill is about 120 tons of slabs per hour. Associated with the slab mill are eight soaking pits each having a holding capacity of 100 tons of ingots. Four of these pits are of the Amsler-Morton square type, the other four being the Salem round type. The weight of slabs produced by this mill varies between 1½ to 4 tons, depending upon the size of the strip required.

In a few American mills operating continuous hot strip the ingot is rolled into the form of a slab, hot sheared to lengths, and the rolling continued to the finished strip without re-heating the slabs, making the slabbing mill a part of the rolling line. This method has the advantage of of conserving much heat, but it can only be adopted successfully when the number of products are limited to the use of very few grades of steel of one type. Because of the wide variety of products and grades and types of steel used, the majority of sheet producers operating continuous hot-strip mills shear their slabs hot, permit them to cool, and then they are re-heated and rolled to the finished product desired. This latter method has the advantage of permitting inspection for surface defects which reduces the amount of finished product discarded, and also facilitates the rolling of small orders requiring different grades of steel. This is the method adopted at the Shotton works.

*Hot-Strip Mill.* After the slabs have been conditioned they are assembled, in their respective grades, adjacent to re-heating furnaces, then they are subjected to various operations designated as heating, descaling, roughing, and finishing. The hot mill at the Shotton works comprises eight stands and the maximum width of hot strip rolled is 53 in. All the mills in the train are of the four. high type, the roughing mill—No. 1 stand—having work rolls 38 in. diameter and backing rolls 49 in. diameter ; the remaining stands have work rolls 22 in. diameter and backing rolls 44 in. diameter. These stands of electrically-driven rolls, arranged in tandem, are provided with supplementary equipment.

While there is usually considerable space between the descaling and roughing train and the finishing stands, the latter, consisting of six stands, are placed at equal distances apart. Each stand of rolls is separately driven and the spaces between the stands are taken up with roll tables made up of individually driven rolls. Devices are arranged to take up an occasional slack from stand to stand. The finishing train is followed by a flying shear, used for cropping the ends of the strip, and then a lengthy run-out table along which the strip is carried to the coiler.

These roughing and finishing stands, together with roll tables, flying shear, run-out table, and coiler, constitute an immense hot-rolling machine, with all parts separately driven but perfectly synchronised to reduce and carry forward the strip, which is continuously increasing in length as it passes through the mill. This mill at the Shotton works is designed to deliver finished-roll strip at speeds up to 1,560 ft. per minute and to a maximum length of strip of about 1,200 ft.

The structure of hot-rolled strip is much finer than that of normalised pack-rolled sheets, and its physical properties are comparable with either normalised or annealed sheets rolled on pack mills. Hot-rolled strip is, therefore, suitable for many applications without heat-treatment. For these purposes it may be used in coils or cut to lengths as sheets. For many purposes, however, because of the need for dimensional accuracy, superior surface finish and particular physical properties for the numerous uses of steel sheet involving stamping, drawing, and other cold-forming operations, the strip is subsequently cold rolled.

*Tandem Cold Reduction Mill.* As previously mentioned, the objects of cold rolling may include precision as to dimensions, control of the hardness or surface finish, or, as it usually does, it may embrace all these, and the amount of reduction in thickness may vary from light skin passes to heavy reductions, effected in three or five passes. At the Shotton works a continuous tandem cold rolling mill is installed having three stands. It is designed for rolling strip up to a maximum of 52 in. wide. Each stand is of the four-high type and carries work rolls 20½ in. diameter with 44 in. backing rolls.

Previously hot-rolled and pickled strip provides the stock for cold rolling and the quality of the finished cold-rolled strip depends largely on the condition of this stock. In the hot rolling of strip for cold rolling, careful attention to detail is necessary to obtain stock of a quality suitable for the purpose. Surface conditions, whether smooth and free from scale or rough and silvery ; rolling temperatures during the final passes, whether high or low, uniform or varied ; and other conditions, have a marked effect on cold-rolling operations as well as on the quality of the final product. Special precautions must be taken to prevent scale being rolled into the surface, otherwise a fitted cold-rolled product would result. For cold rolling, a strip of the proper thickness must be selected to permit sufficient cold working to give the required grain and temper in the finished steel, since cold working of any nature, within certain limits, improves the quality of steel.

Cold-rolled sheets are used for a wide variety of products, and to meet demands for particular types of products the material must satisfy certain specifications. These specifications are not only concerned with dimensional accuracy and surface finish, but also with the quality of the grade of steel rolled. Apart from having the desired mechanical properties the steel should be clean and free from occluded gases which are likely to be released in subsequent treatments to which the products from steel sheet may be subjected. In enamelled goods, for instance, for which sheet is being used to an increasing extent, the release of gas from the steel during the firing of the enamel would be fatal to the success of the operation.

To maintain the right quality in the various grades of steel manufactured careful control is necessary, which involves repeated testing and inspection as normal routine practice. At the Shotton works control of products is effected through laboratories located in various departments, while problems, inseparable from a steelworks, of this type, are handled by the research laboratory of the works.

It has only been possible to give an outline of the facilities for the production of sheet at these works ; much of the subsidiary equipment and plant in the works has been omitted, indeed, no mention has been made of the manufacture of galvanised corrugated sheets for which this Company is noted, but it will be appreciated that, with the installation of the new cold reduction reversible mill, which will give greater flexibility in production, every effort is being made by this Company to assist in meeting the enormous demand for steel sheet of widely varying size and grade.

# Whither Metallurgy?

By J. W. Jenkin, Ph.D., B.Sc., A.R.I.C., F.I.M.

*In a recent address to the Sheffield Metallurgical Association, Dr. Jenkin put the above question, not because he could answer it, but because, as he said in his opening remarks, it is no bad exercise at times to indulge in the art of "imagineering" or wondering "where do we go from here?" On broad lines he traced the development of metallurgy through the centuries and directed attention to some of the modern tendencies. Much of his address was intended only for members of the Association, but by far the greater part will have a wider interest, and it is this part with which the following abridgment is concerned.*

METALLURGY is a word derived from two Greek words, "metallon" and "ergos," meaning simply "metals" and "work." On that simple definition "metallurgy" means just working amongst metals and therefore includes everything from the initial production of the metal to the final product. Although the word itself is adopted from the Greek through the Latin "metallurgia," the Oxford English Dictionary records no instance of its use in this country until some 250 years ago. In 1704, one J. Harris described metallurgy in a text book as "The working or operation upon metals, in order to render them most fine, hard, bright, beautiful, serviceable or useful to mankind."

In this first use of the word there is clearly no restriction to the extraction of metals from their ores, but within a century, namely in the third edition of the Encyclopedia Britannica, dated 1797, we find this phrase, "With others, therefore, we have chosen to restrain metallurgy to those operations required to separate metals from their ores for the uses of life." In view of the state of knowledge at that time this is not very surprising, but in the latest edition of that same noble work we find the following: "Metallurgy, a term formerly defined as the art of extracting metals from their ores, has now a wider meaning. It is applied by modern metallurgists to the structure of metals and of alloys, to their constitution and its relation to their physical properties, and to the thermal and mechanical treatment of metals."

This means, of course, that the term "metallurgy," as the result of the enormous progress of the last three generations, has outlived the restricted meaning formerly applied to it, and reverted to its original and etymological sense of the working of metals. The definition of 1797 refers to it as an art, for then it was little more. To-day's definition recognises that it is a science without being anything less of an art.

### First Materials Used by Man

Clearly the first materials used by man must have been *non*-metallic, because of their very occurrence in the earth. But some metals occur native, and there is no doubt that man's attention was first drawn to metals by the existence of copper, silver, and gold in the uncombined state. He would naturally be attracted by their colour, lustre and relative freedom from corrosion. He must have looked upon them as things of beauty before he learned how to work them. There is evidence, I am told, that long before any Egyptian civilisation metals were in use in Chaldea (the plain of the rivers Tigris and Euphrates between Baghdad and Basra, at the head of the Persian Gulf) although that country contained no mines and no fuel, so the knowledge that metals could be obtained from the earth apart from their native occurrence must have been slowly acquired somewhere else and imported into Chaldea. In those ancient days pigments were also known, and one of them in common use was the green substance, malachite. It has been suggested that when débris containing malachite was dropped amongst the embers of a charcoal fire, the malachite (which is copper carbonate) was first oxidised to copper oxide and then reduced to the metal on roasting with charcoal.

Probably 2,000 years elapsed between the early discovery that native copper could be hardened by hammering and the further momentous discovery that copper could be produced by oxidation and reduction. As we know, ores of different metals often occur together; indeed, although some writers claim that the Greek word "metallon" means "to search after," Pliny (the Roman who perished in the eruption of Vesuvius in A.D. 79 when Pompeii was buried) says that the Greeks before him had used the word "metallon" because metals occurred together "met alla." Certainly it is generally held that bronze, which dominated civilisation for so long that archeologists refer to a Bronze Age, must have been obtainable because copper and tin ores occur together. The biblical reference to "Out of whose hills thou mayest dig brass" refers, of course, to bronze, because the translators of 1611 did not define brass as a copper-zinc alloy as we do to-day.

The metal iron rarely occurs native, so that its discovery was probably much later than that of copper, silver and gold. It is quite likely that iron was found as the result of the same process as copper, such as by the accidental roasting with charcoal of some common pigment like the ochre hematite. The beginnings of metallurgy probably lie in this extraction of several of the then known metals by reduction, that is by heating the ores with charcoal and a limited supply of air.

Probably the next stage was the discovery that some stones that would not give metals in this way would nevertheless do so if heated in air first. That is, they were sulphides that needed oxidation first. Iron sulphide, occurring as pyrites, must have been a difficult one, and may well have led to the invention of bellows. This is ascribed to the Egyptians, who were much more practical than the earlier Chaldeans, and it is likely that the bellows consisted essentially of two goat skins, which

the operator trod on alternately. Similar methods are in use to-day in iron smelting by the natives of the Gold Coast.

While doubtless the Chaldeans worked in iron as well as other metals, the records of Egyptian civilisation are much more clear in practical detail. The Chaldeans were the pioneers of alchemy, and linked the metals with the planets: the sun with gold, Venus with copper, Mars with iron, Jupiter with tin, Saturn with lead, and the moon with silver. The last has persisted in the use of the term "lunar caustic" for silver nitrate. According to this linkage with the planets the best time for extracting the metals or working them was when the appropriate planet was crossing the meridian and especially for two metals when the two corresponding planets were in conjunction. In connection with the more practical knowledge of the Egyptians, it has been said that Moses brought the golden calf into contempt as a false god because he knew how to fuse it with an alkaline earth, probably calcium sulphide.

The association of iron with Mars is of interest in view of two quotations from the Greek and Roman, one of them ten centuries B.C., when Hesiod said that the age of iron was a time when "Sorrow ruled the world, crime was rampant, man delighted in destroying his neighbour, truth was rare, and kindness unknown." About a thousand years later Pliny observed that iron was a minister of war, murder and robbery, but nobly added: "Let the blame for such . . . be brought home to man and not to nature." His words hold good 2,000 years later, for we still readily blame inanimate objects.

The metallurgical knowledge of the Egyptians was in due course inherited by the Greeks, who were of a more philosophical and much less practical turn of mind. The Greeks were ironmasters, and indeed may have discovered iron for themselves, but certainly were not the first to do so. They made no practical advance in methods of extracting iron. Nor did the Romans, who although they knew the value of iron were not particularly skilled in working it. But the Carthaginians were, and Hannibal's victory over the Romans is attributed to the superiority of his soldiers' swords.

## The Philosopher and the Artificer in Early Civilisations

The tendency in these early civilisations seems to have been for metallurgical knowledge to follow two streams, the practical and the theoretical, and the connections between them got more and more unreal. The earliest metallurgists, the Chaldeans, were dreamers, linking metals with the planets, so were the Greeks, who furthered the Chaldean ideas of alchemy; the very word alchemy is said to have been derived from the Greek "chyma" meaning things melted and poured out. The Egyptians, however, reached considerable practical skill: they used iron as early as 4,000 B.C. in the construction of the Great Pyramid at Gizeh. The Assyrians and Babylonians were also practical people; the artificers of the city of Nineveh made very fine hammers, chain armour, and helmets. So metallurgy was wholly a craft, or perhaps I should say an art; it was purely empirical, and Greek and Roman philosophy made no fundamental contribution to its material progress. Those who practised it were artificers. We look upon Tubal-cain as one of the earliest metallurgists mentioned by name: we are told not merely that he was an artificer, but that he was an "instructer of every artificer in brass and iron," so he drew no distinction between the ferrous and non-ferrous!

This early divorcement of the philosopher from the artificer was very disastrous. It prevented the development of metallurgy for literally thousands of years. To the early thinkers, it was a branch of chemistry, and the main activity was the search for the philosopher's stone, that elusive substance that would not only change all base metals into gold, but would confer immortality on the discoverer. That wild-goose chase blinded man for centuries, and he made no progress of an understanding character until various bright intellects pointed out the necessity for making theories fit the facts, and not vice versa.

## Early Progress in Methods and Tools

Although there was practically no progress in the understanding of metallurgy for some 3,000 years, there was, of course, progress in methods and tools, even if there were no fundamental changes. We have a fine legacy of the work of medieval craftsmen, but the limitations on transport and communications meant that work was of local significance, and dependent on local materials.

One of the contributions of ancient Greece was that the conception of a State or civic community grew apace, and it fostered the idea of craftsmanship, the specialist taking the place of the family handyman. Equipment must, of course, have been very primitive: furnaces were just conical holes in sloping ground, and could not be worked unless the wind was in the right quarter. Perhaps the Egyptians reached forging temperatures by lung power, for slave labour was plentiful, later advancing to bellows. The first use of forced draught appears to have been a crude pump, a piston working in a bamboo, in India and Borneo. The early Britons, too, made rough iron weapons and beat their ploughshares into swords in the form of scythes for attaching to chariot wheels. Temperatures could not have been high, and it is likely that instead of ores being reduced by charcoal to the extent of melting the metal, impurities were subsequently squeezed out by hammering.

The most famous product of Ancient Britain, however, was Cornish tin. There is much legendary lore about the trade with the Phœnicians about a thousand years B.C., but although it is referred to by Roman writers, the detail is shadowy and uncertain. Archeological findings, however, do offer some evidence of not much later date. In the course of excavations only 20 years ago near St. Just, in West Cornwall, a series of small smelting pits—i.e., open furnaces, was discovered in a hill castle which other evidence proves to be as old as 300–200 B.C. In one of these pits lay a lump of slag which an assay proved to be largely tin. We can, therefore, safely assume an antiquity of over 2,000 years for the tin industry of Cornwall. For the last 800 years it has been pretty fully documented.

In the case of iron, not even in the Middle Ages was it really melted, in spite of the introduction of the Catalan forge in Spain about 1300, in which an air blast of great regularity was obtained by means of a 25 ft. waterfall. Down the centuries the size of furnaces increased, necessitating side walls and improved blowing arrangements, but the process remained essentially the same, just because it was not understood. For the same reason the methods of extracting the then known metals

at the end of the 17th century were the same in principle as in the early civilisations, and that goes for gold, silver, copper, tin, iron, lead, zinc, mercury and antimony.

There were, of course, advances in the technique of metalliferous mining, for whereas in early history slave labour was employed and there was no thought for the workers' safety, by the 16th century we find much detail in one of the most famous metallurgical treatises : *Libri Duodec'm de Re Me allic'*, by Georgius Agricola, whose real name was Georg Bauer. He was born in Germany in 1494 and studied medicine at Leipzig and in Italy. His series of books appeared in 1561, a few years after his death. He describes the machinery and implements of mining and metallurgy, teaches the art of preparing and purifying copper and iron by means of lead, and describes puddling, in which connection he looks upon steel as a very pure iron. Agricola discovered bismuth, and made the earliest suggestion of blowpipe analysis and flame tests. In one of his books he deals with pumping and ventilation, with illustrations, and with the ailments and accidents to which miners are prone. He must have been impatient of the philosophical school, and must have been a good scientist and thinker as well as a practical man. Two quotations will illustrate this : " A miner, since we think he ought to be a good and serious man, should not make use of an enchanted twig, because if he is prudent and skilled in the nature of signs, he understands that a forked stick is of no use to him." Again, " Man does not need metals to fight with : sticks, stones, fists, heels and teeth would also serve. Metals are not to be condemned, but our own vices such as anger, cruelty, discord, passion for power, avarice and lust." How true 400 years later !

### The Beginning of a Scientific Approach

This was about the time that men began to question the philosophical ideas that had persisted since the time of Aristotle, and within a hundred years we have the eloquent plea of Francis Bacon for the Advancement of Learning in an appeal to facts. This was the beginning of the true scientific method, and in due course men began to wonder what really happened when the metals were extracted from their ores. In Agricola's time the sole use of charcoal as a fuel had greatly reduced Britain's forests, and when in 1558 the Government banned the use of charcoal the effect was to cut down the iron industry to a quarter of its previous size and importance. This however was a blessing in disguise, for it led to the use of pit coal. Dud Dudley pioneered this in 1621, and the owners of woods and the charcoal burners resented his interference with their monopoly and actively opposed him. The barrier to metallurgical progress, however, the lack of understanding of its processes, was not removed until the middle of the 18th century, 200 years ago, when such people as Priestley in Birmingham and Lavoisier in France settled once and for all the mechanism of oxidation and reduction. Their work was done in the interests of chemistry, and it did not have its immediate effect on the art of metallurgy.

### Rapid Advances in Metallurgy

Meanwhile the new learning had paved the way for the Industrial Revolution, which may be said to have begun in Birmingham where Matthew Boulton set up a hardware factory in 1760. It was a hundred years after that, and thus less than a century ago, that with increasing demand the metallurgical industries came into their own. A better understanding of the fundamental physics involved led to progress at an accelerated rate. Metallurgists felt no longer tied to oxidation and reduction processes for extracting metals. They learnt to make and to use metals that had been isolated for the first time within the previous century. Physical metallurgy grew and provided an understanding of mechanical and thermal treatment, of alloying, and all the vast detail of metallurgy as we know it to-day.

There have been few more far-reaching discoveries than the Bessemer and open-hearth processes for making steel, and processes for the production of the light metals by the electrolysis of fused compounds. During the past three generations a greater quantity of metals and alloys has been made than in all previous history, and there has been an enormous increase in the applications to engineering.

Well before the 19th century ended, metallurgy began to outgrow the chemical approach to the subject that had been characteristic of it through the ages, and while chemistry is still one of its fundamental sciences, metallurgy is a borderland of physics, chemistry and mechanical engineering, as well as a country where the practical skill of the craftsman and the scientist's disinterested pursuit of truth are allied in the service of mankind. Unfortunately that service has recently had to be devoted to destructive ends. It is a thousand pities that it seems to take a terrible war—or two wars in our generation—to produce great and rapid advances in a subject that can do so much for the arts of peace, but even to destructive ends many miracles of production would have been impracticable but for the achievements of the metallurgist. One has only to recall the developments in aluminium and magnesium alloys, the co-ordination of steel compositions in the interests of conservation of raw materials, the extraction of magnesium from sea water, new methods of shaping metals and forming and forging them into instruments of war, enormous strides in the application of welding, output figures previously undreamt of, and in particular the development of new alloys that stand up to stresses at extremely high temperatures, without which the gas turbine would have been impossible.

Great things have been done and are being done in our time, and we are amongst the doers. We have within living memory learnt thousands of new facts about metals and alloys and their properties, all acquired by systematic and painstaking scientific research. And yet we are only just beginning to understand why the facts are as they are : our work is still much more descriptive than explanatory. The number of metals known in 1700 A.D. was very few more than in 1700 B.C. ; to-day we have several times as many, and the possible combinations in the way of alloys are legion.

We can say with some assurance that there are no more metals to be discovered, but we cannot say that we understand their behaviour fully ; when we do it is quite likely that we shall achieve more useful properties than we dream of now. Certain it is that metallurgy has outlived its former restrictive sense of the extraction of metals from their ores, and more than justified the etymology of the word. It is a combination of art and science, and has long since attained the dignity of a profession.

### Progress and Research

The astounding metallurgical progress of the last two or three generations can be correctly described as the

results of research. While not in any way belittling the work of those of us who are engaged in production control, in inspection, and in many other activities, it is to research that we look for progress. And it really is surprising how many of us are engaged in research, even if we are not nominally described as belonging to research departments. As metallurgists we are all interested in new ways of handling materials and constantly striving for improvement. We study existing techniques with an eye all the time on the possibility of evolving better ones. What production metallurgist would not seize an opportunity of producing a better article at the same price, or preferably the same article at a lower cost ? While we all have a part to play in technical progress I am not hesitating to say, however, that research must not be confined to the spare-time activity of those whose primary duty lies in production, administration, sales, and the other functions that require an attitude of mind different from the scientific temper of the research man.

Naturally a scientific research metallurgist cannot make progress of ultimate economic value unless he has industrial experience and keeps in the closest daily touch with the other aspects of the industry he seeks to serve. Fortunately the metallurgical industries that we serve are fully alive to the need for putting research into departments that are especially established for the purpose, staffed by personnel trained in research methods, and there is no need for me to preach to the converted. It cannot be too strongly emphasised, however, that research is not the pursuit of a few sequestered scientists tucked away in laboratories that are in danger of becoming remote from practical affairs. Most metallurgists need a laboratory, but it is merely one of their tools.

There is often talk of the enormous time lag between laboratory investigations and their practical application, and we are apt to ascribe that to the conservatism of the British business man. There is possibly another reason in the tendency of some of our scientific friends to look at research through the doorway of a laboratory, but looking inwards instead of outwards. I believe that research is inseparably linked with development, defining the term development as the transition stage between research and the application of the results to commercial advantage. British industry has often been accused of being slow and inefficient in bridging that gap : one reason is, I think, that there has been an insufficient appreciation of the fact that research and development interlock at all levels : they need essentially the same attitude of mind and differ only in the scale of operations. Research departments should be departments of development and research, and the tendency in that direction to-day is agreeably noticeable.

Research is, after all, essentially the intentional seeking of new knowledge. On that broad definition it is as applicable to history as to music, as long as it involves the scientific method. This characteristic method of science is the systematic examination of facts with a view to discovering the underlying principles or generalisations. It means something more than the mere collecting of facts, because it implies the creative thinking required to see the relations between them, and so to devise the crucial experiments necessary to prove a particular theory built around previously known facts. The ultimate appeal is always to experiment : as a famous journalist said in his professional capacity, " facts are sacred, but comment is free."

This scientific method is one in which the laws of evidence are of paramount importance, and it is entirely foreign to the habit of viewing situations through the eyes of sentiment, or preconceived notions, or indeed anything other than the complete facts disinterestedly examined. It is really part of the age-long quest for truth, characterised by a ruthless objectivity. These considerations apply as much to semi-production scale development as to laboratory research. What we need in metallurgical research is men (or, of course, women) with a mental background wide enough to enlist the aid of perhaps several branches of applied science in the solution of any one puzzle. The research metallurgist needs an ability to get evidence that will be both necessary and sufficient, to see clearly the effect of variables, and to weigh the evidence impartially. He knows it is absolutely fatal to try to bluff himself or his fellows. If there is any whitewashing to be done, there is no lack of other people competent and willing to do it.

If a research organisation is to be free from routine worries, with no commission but to sift new ideas and ensure technical progress, it can only be an overhead charge on industry. One cannot make discoveries to order, and no research department can pay a regular dividend. It needs faith on the part of the industrialist, and is doomed to failure unless the man in charge of it enlists the enthusiasm of everybody from the board of directors down to the humblest man in the shops. Your research man must be a dreamer, but must have his feet on the ground and possess a proper sense of proportion, or else he will get scant sympathy from those who have to endure him as an overhead charge. He must never be content with the second-rate, since his recommendations must be unmistakably clear and an error in his findings might mislead a whole industry and prove costly in the extreme.

### Production Metallurgists

It is necessary, of course, to preserve a sense of proportion and to recognise that research is but one facet of industrial administration, whereas metallurgists have their part to play in teaching, in inspection and in process control at every stage, from the mining of the ore through concentrating and smelting operations, in the manufacture of the metals themselves in ingot form, their fabrication into final shape to their behaviour in subsequent service to engineering. Metallurgists may indeed even be salesmen, when I can well imagine that their attitude of mind may differ from that of the research man, but their fundamental metallurgical knowledge is the same.

If a sense of proportion is essential to the research man, it is no less important for the works metallurgist. It is the metallurgist in the works who is responsible for the day-to-day application of the principles of metallurgy to the ever-present problem of increasing the efficiency of British industry.

There are more production metallurgists than research metallurgists, and their work is of immediate practical consequence. The works metallurgist needs a training no less thorough ; it is a mistake to think that the research man is a superior being. The works metallurgist needs rather a different attitude of mind, he is always hedged round by the importance of time and cost, he has to make rapid decisions depending on local and temporal conditions ; he may be the interpreter of the results of research, but he always has to keep a friendly

eye on such departments as labour, costing, estimating and sales. He has to deal with the frailties of human nature, to say nothing of the frailty of certain steels. He needs imagination, and the wider his cultural background the better. He needs a laboratory as one of his tools, but his real work is on the plant. Similarly the research man needs a laboratory, but he also needs to be in the shops, too. Your research man is inclined to wait till things are certain, and when he does it is nearly certain that you will always wait for him. The works metallurgist often has to use judgment, which Bertrand Russell has described as "the art of reaching sound conclusions on inadequate data."

### The Future of Metallurgy

In the face of this overriding importance of the metallurgist being essentially a plant man, it is pertinent to ask, "Whither metallurgy?" in view of the predominant contribution in recent years of the physicist. I believe that the physicist has a greater contribution to make to metallurgical research than, for example, the chemist. This is particularly true of metallurgy in this country because of the much greater extent of the industrial effort that is devoted to the melting, casting, fabrication, heat-treatment, etc., of metals than to their extraction from the ore. I suppose it is true to say that iron is substantially the only metal that is extracted from its ores in this country. The last few years have seen a remarkable application not only of physical methods of examination but also of the fundamental concepts of physics to our understanding of the metallic state.

There is a tendency in some quarters to suggest that the real progress in metallurgy in recent years has been due to people who are physicists by training or to metallurgists who have acquired the fundamental physical knowledge. It is even suggested that there is a danger of metallurgists being displaced from physical metallurgy by physicists and engineers. We now talk of "metal physics" instead of "physical metallurgy." I am all in favour of closer contact between physicists and metallurgists, and of increasing attention being paid to physics in metallurgical training. However, in encouraging this tendency, we must not lose sight of the fact that the majority of metallurgists are not engaged in research work in physical metallurgy. Most of them are concerned with the control of materials in production processes; they are busy in the manifold task of making things work, handling materials in bulk under conditions remote from the laboratory scale of operations.

The great importance of wave mechanics, atomic structure and free-electron theories to the research worker in meta physics should ot offset the need for an ability to appreciate "bulk metallurgy," which, whi e being essentially applied physics, requires more than a nodding acquaintance with mechanical engineering; the closest relative of the metallurgist in practice is the mechanical engineer, who could hardly start work without the metallurgist, but equally the metallurgist's knowledge of materials would be of little avail without the engineer. The practising metallurgist needs some knowledge of several borderland subjects, including industrial organisation and administration. Let us, therefore, welcome any advocacy of the importance of fundamental physics as long as we recognise that most metallurgists are engaged in severely practical affairs rather than in research in physical metallurgy.

One thing that does stand out from a review of current developments and future possibilities is the need for the maintenance of the highest possible standards. This old country of ours is going to have a tough time in the next few years, and the only way to recovery is by every one of us pulling his weight. It is our bounden duty to ourselves, to the teams in which we take part, whether in industry or elsewhere, to the community at large, and to our nation that we make ourselves as competent and as highly qualified as we know how. The race is to the swift and the battle to the strong, and the only hope of improving the standard of living is by sheer efficiency. In metallurgy there is an enormous amount of work to do, and any condoning of inefficient and shoddy work would be to our shame.

This question of the preservation of standards brings me appropriately to a reference to the Institution of Metallurgists, of which I have the honour to be President for the time being. There is no doubt in the public mind that membership of a suitable Institution is a guarantee of a high standard of professional competence and integrity. The last hundred years or more since the Industrial Revolution have seen the formation of many professional institutions with such objects as the establishment of examinations and other means of ensuring the competence of members, the interchange of specialised information, and the advancement of their particular branch of learning. The lawyers, the engineers, the chemists, the architects, and the medical men have for a long time banded themselves together in this way. The metallurgist on the other hand, who practises one of the oldest of the arts and one (or should I say some) of the youngest of the sciences, has until a year ago had no professional Institution that would provide a hallmark of competence in the science and practice of metallurgy.

A generation and more ago when metallurgy was rightly looked upon as a branch of chemistry, the men who studied metallurgy aimed at the Fellowship of the Royal Institute of Chemistry in that branch. As long as the metallurgist was concerned mainly with the art of extracting metals from their ores, he could similarly attain professional standing by becoming a member of the Institution of Mining and Metallurgy, a body which takes great care about admission to its ranks, but is not, I believe, an examining institution, nor is it closely concerned with metals once they have been separated from their ores and refined. In the territory now covered by metallurgy, as described earlier in this story, there are numerous organisations devoted to the dissemination of specialised knowledge, and I need only remind you of the noble work done by the Iron and Steel Institute since 1869 and the Institute of Metals since 1908. These are bodies of international repute; so are the American Society for Metals and the American Institute of Mining and Metallurgical Engineers. None of these bodies, however, pretends to be a qualifying institution, and many of them are by constitution debarred from doing so.

The field of metallurgical organisations is now complete with the formation of a professional body, the Institution of Metallurgists. That this was a sound move is shown by the remarkable number of applications for membership that have been received in the first year. The number of members elected is already over a thousand, and it has been so embarrassingly successful that we have found it impracticable to keep pace with the demand. But there has been no desire to create a large

membership for the sake of being able to quote impressively large numbers. Indeed, the Council has been prompted only by the importance of maintaining a high standard. By reason of the fundamental nature of our work, and the desire that it shall be recognised, it is all the more important that there should be standards for the safeguarding of the public interest. Thus our first object is to establish qualifications that in due time will come to be regarded as the hallmark of competence in both the science and practice of metallurgy.

The metallurgist of the future will know that while the number of available pure metals is limited, his life will not be long enough to do more than scratch the surface of the vast field of millions of possible alloys. While he will be conscious of his professional solidarity he will appreciate that he is but one of a team of workers. The metallurgist of the future is likely to make many contributions to our art; he will be concerned with new methods of fabrication and manipulation, both hot and cold, new methods of generating heat, new ways of examining metals; he will seek and find new criteria of suitability for given service, and new understanding of how metals fail. The metallurgist of the future will realise the enormous contribution that has yet to be made to his subject by his friend the physicist, for until we know more fully why the facts are as they are, we shall not completely realise the latent strengths and other properties possessed by the wonderful class of solids with which we deal.

The metallurgist of the future will recognise, too, that when he can combine his specialised knowledge with commonsense and high personal qualifications, and only then, he will be fitted to play his part in the higher administration of the complex thing we call modern civilisation. It is then that he will ensure the proper appreciation of metallurgy by those so dependent upon it, which means everybody. He will play his part efficiently as a man with a sense of responsibility to his profession and to the public in the severely practical affairs of making things work and raising the standard of living. In the immediate future he has a great task in helping industry towards rehabilitation and in assisting Britain to compete with other highly-developed countries in the export drive that is so essential to our recovery from the ravages of war.

As a scientist, too, he will help to infuse the scientific temperament into those with whom he comes into contact, an attitude of mind born of the first duty of a scientist, to seek the truth. Please do not expect me to answer the question, "What is truth?" for like the elastic limit it seems to vary with the accuracy of one's measuring instruments; perhaps truth is only what those who think they know regard as the nearest approach to reality, but I cannot refrain from quoting from a book that is found in the Apocrypha between the Old and New Testaments: in the first book of Esdras, chapter 4, we read: "As for the truth, it endureth, and is always strong; it liveth and endureth for evermore. Truth is the strength, the kingdom, the power and the majesty of all ages."

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## The Work of The Geological Survey of Uganda

THE fifth of the Imperial Institute's series of lectures on the geology and mineral resources of various parts of the Colonial Empire by visiting senior officials was given to-day by Mr. C. B. Bisset, Deputy Director of the Geological Survey of Uganda.

Laying a logical foundation to the work of his department, Mr. Bisset first briefly summarised the principal geographical aspects of the Protectorate. Uganda, with an area of about 90,000 square miles, is approximately equal in size to Great Britain, and although it lies about the equator, the climate is ameliorated by its general elevation of some 3,000–4,000 ft. Slightly higher country of 5,000–6,000 ft. is found in the south-west, and rather lower country than the general level, down to 2,000 ft., occurs in the north-west. Mount Ruwenzori, with its permanent ice-cap, is the highest point at nearly 16,800 ft., whilst in extreme contrast are the papyrus swamps of the southern half of the country. Communications are on the whole good, though detailed traversing is hindered by most of the difficulties usually found in Central Africa.

Little was known about the geology of the Protectorate before 1919 when the Survey was formed, and although the broad outlines are now delineated, a great deal of work still remains to be done. Mr. Bisset gave an account of the provisional conclusions of the Survey as to the geological history of the country.

Turning to economic geology, the lecturer pointed out that Uganda is not noted for its mineral wealth or production. Little was done about economic minerals in the country until the foundation of the Survey, and the position is also complicated by the ownership of the mineral rights over about one-fifth of the country by native landowners. Geographical location is an adverse factor to mineral exports and there is no local market for useful mineral products.

Gold, tin, columbite-tantalite, wolfram and bismuth, mica, beryl, bismuto-tantalite, phosphate and salt have been produced, whilst occurrences of copper, petroleum, and many other minerals are known in the country. Water is one of Uganda's most important minerals, and more than 600 bored wells have already been installed. In addition, construction of small reservoirs is proceeding. A grant of £279,000 has moreover been made from the Colonial Development Fund for further improvements.

A third of the minerals produced has resulted from discoveries by the Survey; and from the total production of over £2 million of minerals until 1945, the Government has received in rents, fees and royalties, nearly the total cost of the Geological Survey.

Although this is very creditable in itself, Mr. Bisset pointed out that his department has always taken the view that information should be gained as to all useful minerals whether immediate use can be made of them or not. The rapidly changing needs of industry and the experience of two wars had made it evident that any deposit can unexpectedly or urgently become of great importance.

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## Errata

THE Chairman of the British Steel Founders Association is Mr. Charles S. Gill, not Hill as given on page 329 of our October issue. On the same page the Address to which reference is made was delivered by Mr Basil Gray, Chairman of the Main Technical Committee of this Association.

# MICROANALYSIS

CHEMICAL AND PHYSICAL METHODS

APPARATUS METALLURGICAL APPLICATIONS TECHNIQUE

*RECENTLY, while reading the description, in an American Journal, of some newly designed teaching laboratories, we were intrigued by the following: "Laboratory space is provided for 120 in general chemistry, for 36 in analytical chemistry, for 18 in organic chemistry, and for 10 to 15 in physicel chemistry." On the first reading we welcomed this as a praiseworthy realisation that analytical chemistry must take its rightful place with the other branches in teaching laboratories as well as elsewhere. Readers of this column will realise how such a view would appeal to us. Second thoughts, however, led us to the conclusion that there was probably not so much of the progressive attitude as we had first thought. The lack of mention of inorganic chemistry leads us to suspect that probably "analytical" here implies "inorganic and analytical" and simply refers to the more elementary processes which are included as by right in any chemical course in this country. However that may be, we went on to wonder just what the balance of subjects should be. This is an unending source of discussion, and one to which there may be no answer. By this we mean that there may be no "right" and "wrong." It is probably true that from teaching laboratory to teaching laboratory the amount should vary. It is also probably true that few such laboratories pay enough attention to advanced analytical chemistry—to this there are a few exceptions known to us and to be praised. We should dearly like to know the disposition of lectures in the various branches of chemistry in the above American institution.*

# Apparatus for Quantitative Inorganic Micro-analysis

## Part II—Volumetric Analysis

By Ronald Belcher and Cecil L. Wilson

*In the first part of this paper, methods were described for the preparation of simple pieces of gravimetric apparatus in the laboratory, thus enabling the practice of micro methods without the necessity for expensive equipment. Volumetric methods can also be practised utilising home-made apparatus, and a selection of this which will enable orthodox methods to be carried out is here described, and its construction and handling detailed.*

IT is clear that one may adapt volumetric methods to the small scale either by a reduction of the concentrations of the solutions, using macro apparatus, or by a reduction in the size of the apparatus, maintaining approximately the same concentrations of solutions as are used on the large scale.

While the first method can be applied quite widely, and requires no special apparatus, it is limited by the accuracy of determining the end point. This, it is well known, is greatest when working with solutions of moderate concentration. The application of indicator corrections to weaker solutions may ultimately become too inaccurate to be of any value as the strengths are progressively decreased.

In general, therefore, it is perhaps preferable to take the second course. This necessitates the preparation and calibration of small-scale apparatus. However, using such apparatus, even although home-made, it is possible to attain an accuracy, using the macro concentrations of solutions and indicator, comparable with that obtained by conventional methods.

As in the case of filters, a bewildering variety of apparatus exists for volumetric procedures,[1,2] but for basic necessities this can be strictly limited to a small number of types.

### Pipettes

Since it is always possible to carry out a titration using burettes only—if the two solutions must be measured, two burettes can be used—pipettes are not absolutely essential. But the construction and calibration of a simple pipette is by no means difficult.

For elementary work, the easiest form to use is undoubtedly the wash-out type, which avoids any drainage error. Two basic forms of this type whose construction

1 Masters, *Chem. Age*, 1943, **48**, p. 439.
2 Wyatt, *Analyst*, 1944, **69**, p. 81.

needs no description since it is self-evident, are shown in Fig. 1. The Pregl form, at *a*, is filled by suction to the mark placed at the constriction. After the liquid is delivered, the pipette is washed out with a total volume of wash liquid approximately equal to the volume of the solution delivered, but used in three or four portions. The form with the side-arm,[3] at *b*, is easier to handle, since the suction is applied through the side-arm, using

**Fig. 1.—Simple wash out pipettes.**

**Fig. 3.—Miller burette and stand.**

**Fig. 5.—Winteringham burette.**

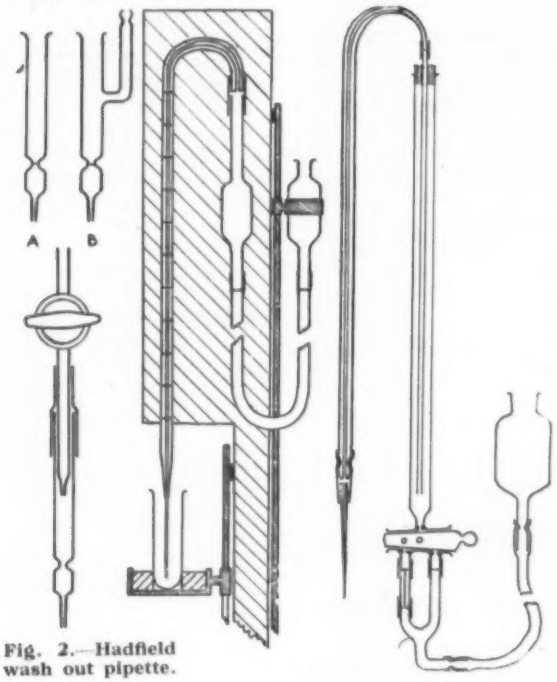


**Fig. 2.—Hadfield wash out pipette.**

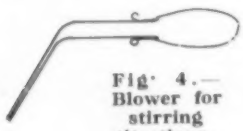

**Fig. 4.—Blower for stirring titrations.**

a rubber tube and mouthpiece, while the washing limb is covered with a finger. Using this form, it is easier to adjust the liquid to the mark.

A slightly more elaborate pipette,[4] but one whose manufacture is still quite straightforward, is shown in Fig. 2. Here a plunger fitted with a tap contains the wash liquid, and at the same time serves to provide the suction required to draw the solution up into the pipette, and the pressure to expel it.

Since wash-out pipettes are calibrated, not for delivery, but for content, they do not need to be calibrated using the solution with which they are ultimately to be employed. Consequently, mercury can be used for calibration purposes, and the calibration may be performed by an ordinary weighing, without the elaborate corrections which would be necessary with a lighter liquid. The calibration error with such a pipette is very low indeed, and as little as 0·001 ml. can be measured readily.

During calibration, it is necessary, of course, to avoid undue warming of either the pipette or the mercury by prolonged contact with the hands.

## Burettes

Burettes may be of two types, vertical or horizontal. Again, only the simplest forms will be described. To avoid the necessity for calibration, it is convenient to construct burettes using the stock 1 ml. straight-stem pipette, graduated in 1/100 ml. However, ordinary glass tubing may be used instead, followed by calibration against an arbitrary scale.

### Vertical Burettes

*Miller burette.* For this form,[5] a pipette is chosen whose tip extends some way below the last graduation, and if necessary a piece of the top is cut off and sealed on to the tip to provide sufficient glass tube to draw out a long jet. The top of the pipette is then bent round in semicircle, as shown in Fig. 3, and is connected by rubber tubing to a levelling system.

The burette is filled by lowering the levelling bulb. Liquid is driven out for the titration by raising the bulb. This continues until the end-point is near, when the bulb is fixed, and the flow of liquid from the burette stops. However, the liquid is held in the burette by the interplay of two sets of forces—hydrostatic pressure, and capillary forces at the burette tip. Although balance has now been attained, the capillary forces may be removed by inserting the tip in the liquid contained in the titration vessel. Further liquid can then flow from the burette, and will continue to do so until the end point is reached, when the titration vessel is lowered again.

Stirring during titration is best achieved by a stream of air bubbles. A jet attached to a blower (or a gas cylinder), or, more simply, the device shown in Fig. 4 may be used for this purpose. The tip of this is inserted in the liquid. The rubber teat is then compressed, and the tip slowly withdrawn while the stream of air bubbles continues. In this way liquid is not drawn up into the device to any appreciable extent.

If a burette of this type is mounted on a wooden stand, it is possible, by a simple arrangement of sliders made from brass curtain rail and brass sheet, to arrange for the levelling bulb and titration vessel to slide up and down easily, so that adjustment of their height is a matter of speed and simplicity. This is achieved by cutting brass sheet to form holders or tables with tabs, which latter are bent round to fit the curtain rail snugly. The rail is attached to the edges of the wooden stand by screws, and separated slightly from the stand by washers to allow for the tabs.

*Winteringham burette.* This form[6] uses a balancing system to expel the liquid from the tip, but has certain advantages which render it very satisfactory in use. A graduated pipette is first drawn out slightly at the tip (Fig. 5) to provide an end to which a tip is attached by rubber tubing. This allows ready replacement of a broken tip. At the other end, the pipette is bent round, and attached to a narrow glass tube, of a bore slightly greater than that of the pipette itself. This tube extends almost to the bottom of a burette-like reservoir, which is fitted with a three-way tap.

The two remaining outlets of the tap are joined by a Y-tube to a mercury levelling bulb, one of the outlets

3 Szent-Gyorgyi, *Biochem. Z.*, 1924, **146**, p. 302.
4 Hadfield, *J.S.C.I.*, 1942, **61**, p. 45.
5 Miller, *Chem. and Ind.*, 1946, p. 26.
6 Winteringham, *Analyst*, 1945, **70**, p. 173.

uses ordinary quill tubing for the junction, while the other employs a very fine capillary tube held in position by rubber tubing. Broken thermometer stem is suitable for this purpose.

A solution is drawn into the burette, using the wide tap-outlet and the mercury levelling bulb. The tap is then closed, and the levelling bulb is raised to bring the mercury in it to a somewhat higher level than that in the central reservoir, thus creating a slight positive pressure inside the burette proper. Delivery of the solution from the burette is now controlled by use of the capillary arm of the tap, the tip of the burette being dipped below the surface of the liquid in the titration vessel. A little experience will show the relative heights of mercury in the central reservoir and levelling bulb which will ensure a satisfactory rate of delivery for any given burette tip, i.e., a satisfactory "sensitivity" for the burette. The burette stand may be fitted with sliders as already described earlier.

### Horizontal Burettes

*Benedetti-Pichler burette.* Horizontal burettes avoid the necessity for a levelling device, since outflow of liquid is controlled almost entirely by capillary forces at the tip. The simplest type, to avoid the necessity for calibration, may, as in the case of vertical burettes, be constructed from a straight-stem calibrated pipette. Alternatively, it may be made from thick-walled 2 mm. bore capillary tubing, bent as shown in Fig. 6. The tip is drawn out to a very fine jet, and is adjusted by trial and error so that when the burette is horizontal, liquid does not flow from it except when the tip is dipped under a liquid, or is touched to a wet surface. Thus capillary force only holds the liquid in the burette, and when this is affected the burette discharges. To avoid drainage errors, a rate of travel of the meniscus in the horizontal tube of 15 mm./min. should be aimed at.

Fig. 6.—Benedetti-Pichler burette

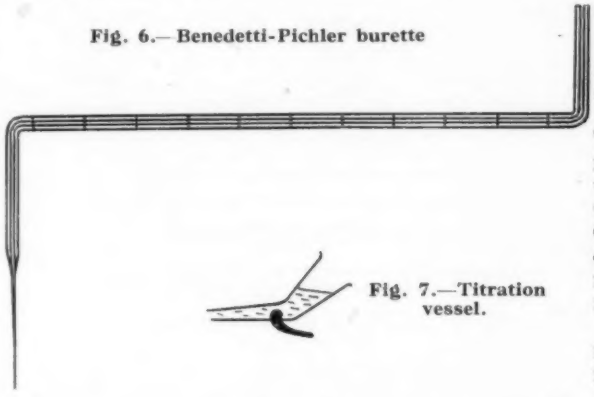

Fig. 7.—Titration vessel.

Such a burette is mounted on a swivelled board, so that the rate of flow can be altered slightly if necessary. However, two points must be observed in use. For any set of determinations a constant rate of flow must be maintained, and in reading the meniscus the board must always be returned to a constant setting. For titration, of course, the tip of the burette is dipped below the surface of the solution being titrated.

If an ungraduated burette is made, this is mounted over a millimetre scale, and must be calibrated. A short length of mercury, say 10 mm., is drawn in, and the variation in its length observed throughout the portion of the burette which is in use, that is, setting one end of the thread at the 1–, 2–, 3– . . . cm. marks. A calibration curve is constructed from these observations. Two of these burettes may then be cross-standardised, using solutions of known strength, and it is not necessary to know the absolute volume delivered by the burettes.

### Titration Vessels

As the simplest expedient, small test-tubes (total capacity 2–5 mls.) or centrifuge cones may be used. These should be chosen so as to give as great as possible depth of solution through which to observe the indicator colour.

An alternative form of vessel is shown in Fig. 7. This is made by bending a tube shaped like a centrifuge cone, to which two small legs made of glass rod have been fused so that the vessel is stable when set on the bench. The indicator colour is observed along the greatest depth of liquid, that is, obliquely.

### Titration procedure

One or two general points may be added concerning the use of burettes such as those described in this article.

Graduated microburettes should be read by means of a lens, and it should be possible to do this to 0·001 ml. Ungraduated burettes, that is, those placed on a millimetre scale, should be read with approximately the same degree of accuracy, say to $\pm 0{\cdot}2$ mm.

The rate of flow of liquid in a burette should be small This follows from the fact that the burette must be read immediately after delivering liquid. Time for drainage to take place would give rise to appreciable errors through evaporation of solution at the tip of the burette Consequently, a delivery rate which will not require drainage time is essential. It is recommended that this rate should lie between 10 and 20 mms. movement of the meniscus per minute.

Before starting a titration, any surplus liquid is removed from the burette tip by wiping with a slip of filter paper.

Since it is advisable that the concentration of indicator in these titrations should approximate to that in macro titrations, it will usually be found that where possible it is best to add the required amount of indicator to the standard solution when it is made up.

For titration on this scale, the volume relations remain similar to those customary on the macro scale. Thus, when using a 50 ml. burette it is usual to take 25 ml. of solution for titration. In the same way, use of a 1 ml. burette would imply commencing with about 0·5 ml. of solution, whether delivered from a pipette or from a burette.

To give most satisfactory results, too, the length of the "reading" portion of a burette using a millimetre scale should lie between 20 and 40 cm., thus retaining the reading accuracy of an ordinary burette. The bore should lie between 0·01 and 3·5 mm. if a horizontal burette is being constructed.

The following Table will help in the selection of suitable tubing.

| Bore of tubing mm. | Approx. volume (mls.) contained in a length of 10 cm. | 20 cm. | 40 cm. |
|---|---|---|---|
| 1 | 0·075 | 0·15 | 0·3 |
| 2 | 0·3 | 0·6 | 1·2 |
| 3 | 0·75 | 1·5 | 3·0 |

[1] Rybbinette and Benedetti-Pichler, *Mikrochemie*, 1942, **30**, p. 15.

The authors contend that in many instances microchemical practice need not wait upon apparatus supply. It is hoped that these articles may convince readers that the gravimetric and volumetric fields of analysis are particularly good examples of this thesis.

## A Filtering Apparatus

By F. Hudswell

THE apparatus described in the following is designed for semi-micro and micro work, and is especially useful for collecting filtrates directly in a graduated flask. When a solid is being extracted with a solvent from a sintered glass crucible, it is always advisable to repeat the extracting, drying, and weighing process, and it frequently happens that a third extraction is necessary to check the constancy of weight of the extracted residue. The advantages of using a graduated flask in such circumstances are apparent.

The adaptor shown (Fig. 1), which allows of gentle suction being applied, and which fits over the stem of the flask and carries the sintered glass filter, is easily constructed by any average glass-blower. When in position on the flask, it rests on a rubber cone (such as is used for holding a Gooch crucible). The cone A, is first slotted over the neck of the flask with the greater diameter at the bottom. At the upper end of the adaptor the short projection of glass tube B, is surrounded with closely fitting rubber tubing C, the latter being slightly longer in order to form a cushion for the filter, and to avoid leakage of air between the two glass surfaces. The internal projection of B serves to locate the adaptor symmetrically around the neck of the flask, and to protect the stem of the filter.

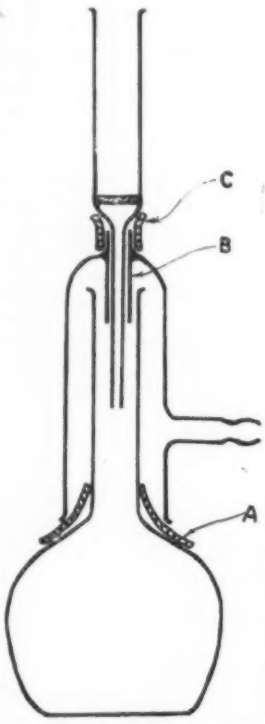


**Fig. 1.—Filtering Adaptor in position.**

It will be found that when gentle suction is applied by means of a filter-pump there is no leakage of air, the rubber fittings holding the glass parts snugly together. It is emphasised that suction should only be slight to avoid undue pressure differences inside and outside the flask. In the author's experience, also, it is much easier to work with filters of porosity 4 if the suction applied is not too great.

The apparatus may easily be held in a clamp on a retort stand. It is compact, easily cleaned, and eliminates transfer of the filtrate from a collecting vessel (usually a test-tube with a side-arm) to the graduated flask.

Aliquots from the flask are quickly available, after filling to the mark and mixing, and either qualitative or quantitative tests can be carried out.

Fig. 1 is drawn to scale, but it may be found that the dimensions of the glass adaptor need modifying according to the supply of flasks available.

My thanks are due to the Chief Superintendent of Armament Research for permission to publish this note.

# Miscellaneous Microchemical Devices

## VIII—Spot Plates for Individual Tests

By J. T. Stock and M. A. Fill

SPOT plates of the type shown in Fig. 1 are suitable for the performance of individual tests, and are simple and cheap to prepare from odd scraps of opal glass plate. The so-called "21 ounce" variety, which is about 3 mm. thick, is particularly suitable. The glass

**Fig. 1.—Spot Plate for Individual Tests.**

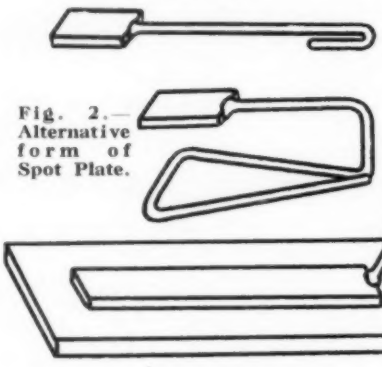

**Fig. 2.—Alternative form of Spot Plate.**




**Fig. 3.—Spot Plate with Identification Label.**

is cut into rectangles, each about 2 cm. × 1 cm. These are sealed on to handles made from 3 mm. diameter glass rod. To avoid burning the fingers during the process, the glass rectangles may be gripped between two pieces of asbestos board. Some varieties of opal glass show a tendency to blacken in the blowpipe flame, so that heating should be cautious. The hook at the end of the handle enables the spot plates to be stored within a covered beaker by hanging them from the rim of the latter.

Numerous modifications in design are possible: for example, the type of spot plate shown in Fig. 2 is suitable for demonstrations or exhibitions. Another form, shown in Fig. 3 is useful when microscopic examination of the spot is desirable. Either a rod-form handle or a tubular one may be provided. The latter form, incorporating a paper identification label, is shown. The label is introduced into the handle before the upper end of the latter is sealed up. This enables plates to be reserved for particular tests. Alternatively, the label may carry a reference number; this is of assistance in avoiding confusion when performing a series of tests.

A few spot plates of this type, but made from clear glass (i.e., from microscope slides) are useful. These may be stood upon any suitable background, such as a block of coloured glass, as shown.

# Heat-treatment of Steel Castings

DURING the war, limitations of weight and concurrent increases of unit strength demanded the use of quenching and tempering treatments for heavy castings which previously were used in the as-cast or as-annealed condition. For medium and low-carbon steels tempered martensite was proved to give the best combination of strength and ductility. In some instances it was sufficient that heavy sections had only an exterior shell that was fully hardened, while in other cases, only fully hardened and tempered parts had an optimum service life. In this connection two papers have recently been published, dealing with the heat-treatment of steel castings. The first paper is concerned chiefly with problems arising in attempts to harden heavy cast steel sections completely and is based on work done at the Battelle Memorial Institute by P. C. Rosenthal and G. K. Manning.[1]

Experiments were first carried out to determine if it was worth while to water quench sections of 6 in. thickness or more, and cooling rates were obtained at the centre and midwall (half-way between the centre and surface) position of plates 3 to 6 in. thickness. The results obtained show a large difference in cooling rate to exist between the centre cooling rate of heavy plates that were air cooled and water quenched. There was only a small difference between the rates of cooling in the midwall position and the rate of cooling in the centre position on heavy water quenched plates. The results also indicated that only a minor temperature gradient existed between the surface and centre of a plate. In a 6 in. plate this did not exceed 130° F. (65° C.) at any particular time during the heating cycle.

As the lowest cooling rate obtainable in the standard end-quench, hardenability test bar was about 4° F. per second at 1,300° F. (700° C.), a special hardenability test bar with a cooling rate ranging from 5° to 1° F. at 1,300° F. was devised. The utility of this air-cooled hardenability test was not so great as anticipated, as when 20 steels of high-alloy content were tested, not one changed more than 2 points Rockwell C between the slowly-cooled and fast-cooled end of the test bar, and only one steel exhibited a conspicuous change in microstructure within the length of the bar. As the microexamination proved to be rather inaccurate, because of the difficulty of distinguishing between martensite and low temperature bainite, more useful information was obtained from the isothermal transformation diagrams of the steels. Curves were obtained showing the influence of manganese, molybdenum, chromium and nickel on the beginning of isothermal transformation in several heavily alloyed steels, Table 1, and indicated that the generous use of alloys led to ready suppression of transformation within the pearlitic range, but was much less effective in suppressing transformation within the bainite range.

TABLE I. ALLOY STEELS USED.

| No. | C | Mn | Ni | Cr | Mo |
|---|---|---|---|---|---|
| 1 | 0·30 | 1·54<br>2·50 | 0·97 | 2·05 | 0·43 |
| 2 | 0·30 | 1·52<br>2·02 | 1·11 | 0·66 | 0·55 |
| 3 | 0·33 | 1·49 | 2·07 | 0·67 | 0·50<br>0·95 |
| 4 | 0·31 | 2·02 | 1·11<br>1·80 | 0·66 | 0·55 |
|  | 0·25 | 1·49 | 2·05<br>3·99 | 0·69 | 0·54 |

From the tests carried out it appeared that by adding generous amounts of alloying elements, a composition of sufficient hardenability, suitable for any given section, could eventually be obtained. Three factors, however, tended to limit the amount of alloying elements that might be used to advantage. First, as the amount of alloying elements was increased, the tendency of the steel to crack during quenching was increased; second, the susceptibility of the steel to temper brittleness was, in general, increased; and third, the use of large amounts of alloys led to increased difficulty in obtaining structures that were readily machineable. Where alloys were reduced as a result of such limitations, a 100% martensite structure might not always be obtained on quenching and there might be a partial transformation to bainite without a large deterioration of mechanical properties.

In the second paper, dealing with the effect of heat-treatment on steel castings, E. E. Howe[2] first considered the mechanics of steel solidification and the homogenisation of the resulting structure by heating the steel to a sufficiently high temperature where diffusion of the various elements can take place. Selection of the correct temperature and length of time required to secure the desired results is governed by the austenitising and grain size changes which take place on heating. The austenitising temperature, being a function of the upper critical temperature on heating, is predetermined on a basis of a slow heating rate, and the length of time at the austenitisation temperature is governed by the composition, shape and volume of the casting.

To determine the effects of heat-treatment on cast steel, mechanical tests were carried out on a steel containing 0·30 to 0·40% carbon, 0·60 to 0·90% manganese, 1·5 to 1·75% nickel, 0·40 to 0·65% chromium and 0·20 to 0·35% molybdenum. Bars of

1 *The Foundry*, 1946, Vol. 74, No. 8, pp. 70-75.

2 *The Foundry*, 1946, Vol. 74, No. 9, pp. 98-99, 194, 195, 198, 199, 200, 202, 204, 206.

TABLE II. RESULTS OF PHYSICAL TESTS ON CAST IN. ROUND TEST BARS.

| Treatment | Tensile Strength Tons per sq. in. | Yield Strength Tons per sq. in. | Elongation % | Reduction of Area % | Brinell Hardness |
|---|---|---|---|---|---|
| As cast | 53·5 | 34·5 | 2·5 | 3·6 | 286 |
| Normalised | 61·8 | 38·8 | 6·5 | 10·4 | 277 |
| Normalised and annealed | 44·5 | 30·0 | 24·5 | 44·9 | 195 |
| Normalised and subcritical annealed | 47·4 | 35·0 | 22·0 | 55·7 | 223 |
| Normalised, annealed, hardened and tempered | 69·4 | 63·1 | 14·5 | 40·7 | 332 |

this steel, $1\frac{1}{4} \times 1\frac{1}{4} \times 6$ in., were subjected to various heat-treatment including normalising, annealing, subcritical annealing, hardening and tempering. Normalising was carried out at 775° C. to obtain solution diffusion of the alloying elements in a short period of time. In annealing, the bars were heated in boxes to 775° C. for 3 hours, then cooled in the furnace to 370° C., and in subcritical annealing, bars were heated to 675 to 700° C. Hardening was carried out at 815° C., followed by oil quenching and tempering at 600° C. The results obtained are given in Table 2.

These results, together with a macroexamination, show that in the as-cast condition, steel lacks homogeneity and is relatively low in strength. Normalising produces some refinement with slight improvement in the physical properties. Box annealing, after normalising, produces further refinement and puts the steel in a condition for rapid machining. Further grain refinement is produced by hardening and causes a brittle condition with high strength. Tempering the hardened steel renders it tougher with comparatively high strength.

# Molybdenum in Cast Iron

By A. E. McRae Smith, M.A., B.Sc.

PRACTICALLY all alloying elements added to cast iron have two main objects. In the first group are elements which are really softeners in that they promote the formation of graphite and normally classed as "graphitisers"; the second group covers elements which induce the formation of carbides and are usually referred to as "carbide" formers. There are degrees of effectiveness in both classes, some having a mild effect while others are much more powerful. Apart from carbon and silicon, the elements most commonly used for their graphitising or softening effect are aluminium, nickel, copper, titanium, and zirconium. The most powerful carbide inducing elements are chromium, vanadium, tellurium, and boron. Manganese can be classed as a carbide-forming element, but its action is very mild.

Molybdenum is usually classified as a mild chill-inducing element, but this does not do it justice, because it is mostly used for its effect on the matrix and not for its effect either to restrain or induce the formation of graphite. In point of fact, there are no conditions under which molybdenum acts even as a mild graphitising agent, but it is the most potent element for generally improving the physical properties of most classes of cast iron.

From the engineer's, as well as the foundryman's point of view, the most important function of molybdenum in grey cast iron is to increase the tensile strength to a marked degree, without having to change normal cupola and other furnace practice, except that the phosphorus content must be really low. This marked beneficial improvement in strength can be attributed mainly to the direct solid solution effect of molybdenum on the matrix. The amounts added range from 0·30–1·2% for most applications.

Apart from this improved tensile strength, varying from 20–50%, molybdenum also improves most of the other physical properties by which the quality of grey cast iron is normally assessed. It acts very much in the same way as it acts in steel. Depth and degree of hardening is much greater and freedom from cracking reduced when molybdenum is present in cast irons which have to be heat-treated.

Quite a number of types of both grey and chilled cast iron are regularly produced in this country on a normal commercial scale. Although molybdenum is often used alone, combinations of nickel and/or chromium with molybdenum have found wider application.

A typical range of composition for straight molybdenum iron castings, varying in sectional thickness from about $\frac{1}{2}$ in. to 3 in. would be as follows:

| | |
|---|---|
| Total Carbon | 2·9–3·2% |
| Silicon | 2·0–2·5% |
| Sulphur | 0·12% max. |
| Phosphorus | 0·15% max. |
| Manganese | 0·7–1·0% |
| Molybdenum | 0·4–0·6% |

A typical range of composition for nickel-molybdenum iron castings would be as follows:

| | |
|---|---|
| Total Carbon | 2·9–3·2% |
| Silicon | 1·6–2·0% |
| Sulphur | 0·12% max. |
| Phosphorus | 0·15% max. |
| Manganese | 0·7–1·0% |
| Nickel | 1·4–1·6% |
| Molybdenum | 0·30–0·40% |

For really massive and lump castings, *e.g.*, press and forging die blocks, forming tools and the like, a small addition of chromium ensures that the material will be close-grained in really heavy sections, and a typical composition in this class would be as follows:

| | |
|---|---|
| Total Carbon | 2·9–3·2% |
| Silicon | 1·4–1·7% |
| Sulphur | 0·12% max. |
| Phosphorus | 0·15% max. |
| Manganese | 0·7–1·0% |
| Nickel | 1·8–2·0% |
| Chromium | 0·4–0·8% (according to the mass of the castings). |
| Molybdenum | 0·4–0·6% |

For certain applications—*e.g.*, engine liners, press sleeves, dies, large gear blanks, cams, etc., where wear resistance, in comparatively heavy section was considered the most important property apart from high strength, chromium-molybdenum compositions were used. A typical example, for castings from say 1½–4 in. general sectional thickness, would be as follows

| | |
|---|---|
| Total Carbon | 2·9–3·2% |
| Silicon | 2·0–2·4% |
| Sulphur | 0·12% max. |
| Phosphorus | 0·15% max. |
| Manganese | 0·8–1·2% |
| Chromium | 0·5–0·7% |
| Molybdenum | 0·6–0·7% |

During the war years Reese and Flinn, in U.S.A., published a very interesting account of their work on nickel-molybdenum cast irons, in which they established a commercial process by which they could obtain cast irons exhibiting "acicular" structures in the as-cast condition, and proved that at least for low-carbon cast irons (2·50% carbon) such castings possessed superior properties to similar castings having the more normal pearlitic structure.

Depending on the mass of the castings being produced, the nickel content will vary between 1·0 and 6·0% and the molybdenum content between 0·2 and 1·5%, but for the majority of castings being produced in acicular cast iron in this country, it is understood that the nickel content will fall within the range 1·50 and 3·50% and the molybdenum content is usually relatively constant at 0·70–0·90%.

Apart from its higher strength, acicular cast iron, probably on account of its unusual matrix structure, possesses much greater toughness and resistance to shock than is the case with pearlitic types of high-strength cast iron, even those containing molybdenum.

Acicular cast iron is being used successfully for such applications as diesel, petrol engine, and compressor crankshafts, camshafts (flame hardened on cams) press and forging dies, and a wide variety of applications for highly-stressed components, where cast iron is used for its rigidity, damp-

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ing capacity (or power to absorb vibrations), wear resistance, self-lubricating properties and the like, and where a non-ductile material will prove satisfactory.

For certain applications engineers and plant manufacturers require castings which will not readily "map line" or "crazy crack" when used under conditions where they are subjected to repeated severe thermal shock—*e.g.*, moulds for the die-casting of copper and other non-ferrous alloys, rollers, and similar devices for the processing of sheet glass, moulds and shapes for glass, ingot, strip, and similar moulds for casting copper alloy billets, special type of brake drums, clutch plates, etc.

It has been well known for many years that high-carbon cast irons containing much graphite are more serviceable for such applications than low-carbon cast irons containing a comparatively small amount of graphite. The extra graphite present in the high-carbon cast irons, all other things being equal, allows them to accommodate their shapes to volume changes, without rupture of the surface, as seen when castings fail prematurely by "map lining" and "crazy cracks."

Experience has recently proved that these high carbon cast irons can be greatly strengthened and provided with close-grained structures, by suitable additions of nickel, chromium, and molybdenum. The latter element, in particular, helps to give extra strength to the matrix and the nickel and chromium give the desired closeness of grain and good finish.

A typical compositional range for most castings would be as follows:

| | |
|---|---|
| Total Carbon | 3·5–3·8% |
| Silicon | 1·4–2·0% (according to sectional thickness) |
| Sulphur | 0·12% max. |
| Phosphorus | 0·10% max. |
| Manganese | 0·6–0·9% |
| Nickel | 1·5–2·0% |
| Chromium | 0·3–0·6% (according to sectional thickness) |
| Molybdenum | 0·7–0·9% |

In recent years molybdenum has taken a prominent place as an alloying element for improving the quality of most types of rolls for the metal-rolling industry. Its usefulness has been proved not only in chilled rolls but also in grain rolls and "indefinite chill" rolls. In combination with chromium and/or nickel it has found wide application. To give an example, for definite chill rolls, where higher strength in the neck is desired, without sacrificing hardness and depth of chill, a typical composition would be as follows:

| | |
|---|---|
| Total Carbon | 3·0–3·3% |
| Silicon | 0·5–0·6% |
| Sulphur | 0·08% max. |
| Phosphorus | 0·10–0·20% |
| Manganese | 0·20–0·40% |
| Nickel | 4·0–4·5% |
| Chromium | 1·2–1·4% |
| Molybdenum | 0·3–0·5% |

For a typical grain roll a suitable composition would be:

| | |
|---|---|
| Total Carbon | 2·5–2·7% |
| Silicon | 0·9–1·1% |
| Sulphur | 0·08% max. |
| Manganese | 0·3–0·4% |
| Phosphorus | 0·10–0·20% |
| Nickel | 1·0–2·0% |
| Chromium | 0·5–0·8% |
| Molybdenum | 0·4–0·5% |

The above compositions are merely given as examples, but there are many others where up to 0·5% or more molybdenum is added for its strength-giving, and grain-refining properties.

The production of molybdenum-contained cast iron is discussed, and methods are given for adding the molybdenum to the cast iron.

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# The Effect of Combined Stresses on the Mechanical Properties of Steels between Room Temperature and —188° C.

By D. J. McAdam, G. W. Geil, and R. W. Mebs

IT has been shown by the authors in previous papers[1,2,3] that the technical cohesive strength of a metal, *i.e.*, its resistance to fracture, is a function of the three principal stresses, and that plastic deformation and temperature affect the technical cohesive strength about as much as they affect flow stress. In this paper, attention is given to stress combinations that are produced by tensile tests of notched cylindrical specimens, and results are given for tensile tests with various notches, at several temperatures between room temperature and –188° C. As a notch in a specimen under longitudinal tension tends to cause both transverse radial tension and stress concentration, the results show the influence of both these variables on the mechanical properties of steel. A study is therefore made of the simultaneous influence of the stress combination, stress concentration, and low temperature in causing brittleness.

In the investigation deep notches were used so as to minimise stress concentration and reveal the effect of the radial stress ratio. The notches were all of the same depth, but the notch angle varied between 170° and 45° and the root radius varied from 0·01 in. to 0·08 in. The principal metals used for the tests were carbon steels. A 0·11% carbon steel and a 0·42% carbon steel were tested in the annealed condition. Cylindrical rods of cold-drawn 0·12 and 0·57% carbon steels were given additional cold work by being extended in a tensile machine until they began to contract locally. The results obtained with these steels were supplemented with results obtained with a few other carbon steels and some alloy steels and comparisons were also made with results obtained from non-ferrous metals.

A description of the metals tests, together with details of their heat-treatment, is given in Table I.

In determining the influence of notch angle and root radius on strength and ductility of steels at various temperatures, the transverse radial stress and stress concentration induced by a notch and their general effect on mechanical properties were first determined, and the results given in diagrams revealing the influence of notches on yield stress, ultimate stress, elongation, and true breaking stress. It was found that both the radial stress ratio and the initial stress concentration increased continuously with decrease in the notch angle and in the root radius. The increase of the radial stress ratio with the decrease of the notch angle tended to cause a continuous rise of a curve of yield stress or ultimate stress. The course of a curve of breaking stress was the resultant of three variables dependent on the notch angle. Two of these were the same variables that affected the ultimate stress and the third variable was the ductility which decreased continuously with decrease in notch angle.

The tensile properties of carbon steels as affected by notches and low temperature were next determined. Diagrams were obtained showing the effect of notches at room temperatures –78°, –128°, and –188° C. on the mechanical properties. At low temperatures the influence of stress concentration became prominent in the curves of ultimate stress and in the curves of breaking stress. Stress concentration manifested itself in the descent of the

From *Proc. Amer. Soc. Testing Mats*, 1945, Vol. 45, pp. 448–481.

1 *Metallurgia*, 1944, Vol. 30, pp. 59–60.

2 *Metallurgia*, 1944, Vol. 31, pp. 106–107.

3 *Metallurgia*, 1945, Vol. 31, pp. 218–219.

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TABLE I—MATERIALS USED AND THEIR HEAT-TREATMENT.

| Metal | Composition % | | | | | | | | Mechanical Treatment | | Heat-Treatment | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | Mn | Ni | Cr | Cu | Sn | Zn | Other Elements | Method | Original Rod Dia. In. | Tem. °C. | Time held Hr. | Cooled in | Tem. °C. | Time held Hr. | Cooled in |
| High-purity Iron | <0·001 | Nil | Nil | — | <0·002 | — | — | 0·001 Si | As forged | 0·750 | 928 | 1 | Furnace | | | |
| 0·029% Carbon Steel | 0·029 | 0·42 | — | — | — | — | — | — | As forged | 0·875 | 928 | ½ | Furnace | | | |
| 0·11% ,, | 0·11 | 0·84 | — | — | — | — | — | — | Cold-rolled | 1·000 | 900 | 1 | ,, | | | |
| 0·12% ,, | 0·12 | 0·82 | — | — | — | — | — | — | ,, | 1·000 | 928 | 1 | ,, | | | |
| 0·12% ,, | 0·12 | 0·44 | — | — | — | — | — | — | Hot-rolled | 1·125 | 928 | 1 | Water | 482 | 1 | Air |
| 0·20% ,, | 0·20 | 0·42 | — | — | — | — | — | — | ,, | 1·125 | 886 | 1 | ,, | 482 | 2 | Air |
| 0·23% ,, | 0·23 | 0·51 | — | — | — | — | — | — | ,, | 1·125 | 900 | 1 | Furnace | | | |
| 0·42% ,, | 0·42 | 0·60 | — | — | — | — | — | — | ,, | 1·125 | 841 | 1 | ,, | | | |
| 0·57% ,, | 0·57 | 0·20 | — | — | — | — | — | — | Cold-worked | 1·125 | 791 | 5/6 | ,, | | | |
| 3·3% Nickel Steel .. | 0·34 | 0·72 | 3·30 | — | — | — | — | — | Hot-rolled | 0·625 | 791 | ¼ | Oil | 537 | 1 | Air |
| 13% Cr, 2% Ni Steel | 0·09 | 0·48 | 2·06 | 13·3 | — | — | — | — | ,, | 0·875 | 670 | — | Furnace | | | |
| 17% Cr, 2% Ni Steel | 0·11 | 0·70 | 1·63 | 17·4 | — | — | — | — | ,, | 0·625 | 950 | ¾ | Air | 730 | 1½ | Furnace |
| Cr-Ni-Mo Steel .. .. | 0·47 | 0·80 | 1·80 | 1·04 | — | — | — | 0·22 Mo | ,, | 0·625 | 785 | — | Furnace | | | |
| Oxygen-free Copper .. | — | — | — | — | 99·97 | — | — | — | Cold-rolled | 0·875 | 426 | 5 | | | | |
| Copper-Tin Alloy .. | — | — | — | — | 95·8 | 3·9 | — | — | Cold-drawn | 0·750 | 65? | ¼ | | | | |
| Brass .. .. .. .. | — | — | — | — | 61·6 | — | 35·11 | — | ,, | 0·875 | 651 | ½ | | | | |
| Monel Metal .. .. | 0·18 | 0·94 | 69·07 | — | 28·46 | — | — | 1·24 Fe | ,, | 0·875 | 765 | 1 | Water | | | |
| Nickel .. .. .. .. | — | — | — | — | — | — | — | — | Hot-rolled | 1 × 1 | 765 | 1 | Air | | | |

curves as the notch angle was decreased below a certain value, which tended to increase as the temperature was lowered, and the descent of such curves at a decreasing rate was in accordance with the variation of the stress concentration with the notch angle. Although the notch angle at which stress concentration became prominent ranged only from 120° to 150°, the corresponding ductility decreased when the temperature was lowered from –78° C. to –128° C. and decreased to less than 1% when the temperature was lowered to –188° C.

The variation of the strength and ductility of annealed and cold-worked steels with temperature was determined by testing the 0·11% carbon annealed, the 0·12% carbon cold worked, and the 0·42% carbon annealed steels. The yield stress and ultimate stress curves for unnotched specimens increased with decrease in temperature to –188° C., and in the former the rate of increase was rapid. The corresponding breaking stress curves increased to –128° C. then descended to a minimum at 188° C. Each curve for notched specimens increased at an increasing rate with decrease of temperature to a certain value dependent on the notch angle and then decreased. The smaller the notch angle the higher the temperature at which descent began.

The influence of composition and heat-treatment on the variation of the strength and ductility of metals with temperature was obtained by testing non-ferrous metals and steels differing in composition and heat-treatment. Yield stress, ultimate stress, and breaking stress curves of unnotched specimens of the three non-ferrous metals and the nickel-chromium steel showed the strength indices for each of these metals to vary linearly with temperature and to increase with decrease of temperature. With decrease in temperature the ductility of each of the non-ferrous metals either remained unchanged or increased, while that of the nickel-chromium steel decreased considerably. In general, heat-treated steels showed an increase in yield stress, ultimate stress, and breaking stress with increase in temperature, but both heat-treated carbon and alloy steels showed some loss of ductility with decrease in temperature. The yield stress line for annealed iron, annealed carbon steels, and low alloy steels curved upwards at both ends from the line representing the linear variation of yield stress of a normal metal, while the lines of yield stress for the highly alloyed alloy steels were practically straight.

# Heat-treatment of High-speed Steel

## By Morris Cohen and Paul Gordon

HIGH-speed steels consist of ferrite complex alloy carbides in the annealed condition, they are austenitised just below their melting point for hardening, and they are tempered to achieve secondary hardness.

It is general practice to carry out the hardening in two steps. First, pre-heat between 1,300° and 1,650° F. Second, soak at a temperature somewhat under the fusion point of the steel, to produce solution of the carbides in the austenite, and prepare the austenite for proper transformation during the subsequent cooling. Important considerations limit the temperature and time of austenitising, the chief of which is austenite grain size, which should remain fine for toughness and strength. In applications where brittleness is not a factor, high austenitising temperatures are advantageous in improving cutting efficiencies through the attainment of higher hot hardness.

Discussing methods of cooling from the austenitising temperature the authors state that it is common practice to hot-quench high-speed steel into the range of 1,000° to 1,200° F. as a part of the hardening operation. Grain-boundary carbide precipitation can be avoided by keeping the temperature of the hot-quenching bath below 1,200°. Conditions for the isothermal annealing of tungsten and tungsten-molybdenum steels are described and microstructures of these steels are shown. The bainite transformation is discussed.

Characteristics of the austenite-martensite reaction are described. This occurs primarily during cooling rather than isothermally like the spheroidite and bainite transformations. If the 18-4-1 steel, for example, is quenched to a temperature of 300° F., the decomposition of austenite begins at 420° F., proceeds as long as the cooling is continued and stops at 300° F. At this temperature the steel contains about 40% martensite.

Normal hardening of high-speed steel seeks to cool the steel from the austenitising temperature in such a way as to avoid decomposition in the two upper ranges of austenite activity and to allow only transformation of

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the austenite into martensite. This is usually accomplished either by oil-quenching or by pre-quenching into a molten bath at, say 1,100° F., and then cooling in air to room temperature. At room temperature the steel consists of untempered martensite, undissolved carbides, and 15 to 25% of retained austenite. The structure is hard (64 to 66 Rc), but is unstable, brittle, and highly stressed. Tempering must alleviate these undesirable characteristics without destroying the hardness.

The reactions which occur on tempering at various temperatures are discussed in detail. The specified times of ½ hour of tempering for maximum secondary hardness and 2½ hours for complete transformation of the retained austenite in 18-4-1 hold only for a tempering temperature of 1,050° F. Changing the tempering temperature changes the rates at which the tempering phenomena occur. Curves are given to illustrate how these rates depend on the tempering temperatures of 950°, 1,000°, 1,050°, 1,100°, and 1,125° F.

The complexities of the hardening treatment with respect to retained austenite are discussed. Arresting the hardening quench at temperatures above 80° F., even when followed by normal tempering, leaves not only appreciable quantities of retained austenite, but also much larger quantities of untempered martensite. Reducing the amount of retained austenite by tempering at 1,250° produces a marked decrease in hardness. A study of multiple tempering was made. This demonstrates that either double or multiple tempering for a cumulative time of 2½ hours at 1,050° F. after quenching to 225° F. results in essentially complete decomposition of the retained austenite. This method of tempering entails no large loss in hardness. An improvement in strength of almost 50% over that obtained by single tempering is possible. This corrective action of multiple tempering applies only to steel whose hardening has been arrested above room temperature. The fact still remains that the highest strength values are secured when the quenching-bath temperature is as low as possible.

Interrupted cooling from the tempering bath results in an improvement in mechanical properties by as much as 10% in transverse strength, 100% in transverse ductility, 10% in shear strength, 45% in torsional ductility, and 120% in torsional toughness. In cooling steel during the hardening quench, austenite transformation continues when the cooling is extended to sub-zero temperatures (–310° F.). Study showed that for 18-4-1 steel, hardened at 2,350° F., cooling lower than –150° gives no further austenite decomposition.

The rate of cooling is without influence, as is also the number of sub-cooling cycles, and the length of time at low temperature, assuming the specimen is held at low temperature long enough to have attained the desired temperature. Various aspects and modifications of the sub-zero cooling are discussed. Subsequent to this cooling, a tempering treatment must be given. It has been shown that on tempering below about 950° F. (one hour), the hardness and strength are greater in the cold-treated steel than in the normally hardened steel, but the reverse is true if the tempering temperature is above 1,000° F. The proportional limit of the cold-treated steel is greater and the plasticity less than the normally hardened steel regardless of the tempering temperature.

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## Antimony

LIKE tungsten, tin, and copper, antimony is a "key" metal, an essential metal difficult to replace by any other. It possesses a number of distinct physical and chemical properties that make substitution well nigh impossible. Thus, for example, contrary to the behaviour of metals in general, antimony, on passing from the liquid to the solid state, expands; other metals contract. This property is taken advantage of in type metal, an alloy of antimony, tin, and lead. Other commercially important antimony alloys are babbitt and antifriction metals, in addition to the 9% antimonial lead alloy for storage battery plates.

In nature antimony usually occurs as sulphide, stibnite, $Sb_2S_3$, disseminated in siliceous rocks and generally associated with quartz. Of secondary commercial importance are the double sulphides such as pyrargyrite ($3\,Ag_2S.Sb_2S_3$), tetrahedrite ($4\,Cu_2S.Sb_2S_3$), and others. Although antimony is very widely distributed in the earth's crust, the three chief producing countries are China, Mexico, and Bolivia. Radical changes have been taking place in the world's antimony trade in the past few years. World production is approximately 30,000 tons per annum.

Numerous researches on antimony have been published, but on account of the unique character of the metal, as to both physical and chemical behaviour, further researches are to be encouraged. Particularly timely would be a thorough investigation of the metal as applied to vacuum tube technology. It is also suggested that a simpler and more efficient method, preferably an electro-metallurgical one, of recovering the metal from low-grade ores is badly needed.

From the *Bull. Electrochem. Soc.*, July, 1946.

## Studies on the Polymorphic Transformation of Selenium

By C. A. Escoffery and S. Halperin

THE various allotropic modifications of selenium are reviewed. The phase change from amorphous to crystalline hexagonal form and the effect of heat-treatments in the manufacture of selenium rectifiers is discussed. Two temperature zones are of paramount importance: one in the vicinity of 100° C. for optimum nucleation, and the second one close to the melting point (ca. 217° C.) for maximum crystal growth. It is shown that selenium will undergo grain growth displaying micro-structures similar to that encountered in metals. Very large grains can be obtained by prolonged heat-treatment. The phase change from the hexagonal to the amorphous form of selenium is also considered. Grain growth is very rapid just below the "melting point." The grains, which enlarge in cross-section at the expense of their neighbours, have a characteristic robin's egg blue colour under polarised light microscopy. At a further stage in their development, these polygonal grains elongate into biconvex-shaped grains surrounded by the amorphous phase, and then gradually dissolve and lose their identities. Photomicrographs of selenium at various stages of grain growth are presented.

From *Electrochem Soc.*, preprint 90-57.

**In order to assist readers who are seeking facts built up in any particular field of enquiry within our field, arrangements have been made with a London research consultant to undertake the compilation of bibliographical and literary research surveys. Although obviously depending upon the nature of the investigation required, the fees for this service will be low. Limited enquiries should be made through *Metallurgia* offices, addressed to the Editor.**